DEVELOPMENT OF A SUITABLE PROCEDURE FOR SIMULATING AGING
EFFECTS OF HOT-MIXING ON MODIFIED ASPHALTS FOR USE IN SUPERPAVE
BINDER SPECIFICATIONS

By

OKAN SIRIN

To my past, present, and future family members.

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By

Okan Sirin

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Chairman: Mang Tia
Major Department: Civil Engineering

This research study was conducted to investigate the feasibility of the Rotavapor

apparatus for simulating short and long-term aging of conventional and modified

asphalts. Preliminary evaluation of the Rotavapor apparatus indicated that the

temperature variation within the oil bath was a major source of variation of this aging

procedure. To improve the temperature uniformity within the oil bath, a well-insulated

box was constructed. A full factorial experiment was also conducted to evaluate the

performance characteristics of the Modified Rotavapor aging apparatus with the insulated

covering.

Extraction of rubber asphalt binders from rubber asphalt mixtures was also

investigated in this study. Better understanding of this process was achieved from this

investigation.

Further improvement on the Modified Rotavapor aging process was made by replacing the standard distillation flask with a Morton flask. The asphalt binders selected in this part of the study include AC-20, AC-30, AC-30+5%CR and AC-30+12%CR. These asphalt binders were aged in the Modified Rotavapor aging process using different testing configurations. The same asphalt binders were also subjected to the TFOT and RTFOT processes. Characterization of these aged asphalt residues were made by performing the following tests

1-) Penetration test at 25 °C,

2-) Brookfield viscosity test at 60 °C

3-) Dynamic Shear Rheometer Test at 25 and 64 °C

4-) Mass change determination

Test results indicate that the Modified Rotavapor aging procedure using 163 °C process temperature, 200 g sample size and 160 minutes duration produces aging severity similar to the TFOT process. However, the aging severity of the RTFOT process was achieved in the Modified Rotavapor aging process at the test configuration of 163 °C, 200 g and 210 minutes.

The applicability of the Modified Rotavapor aging process was also evaluated by grading two asphalt binders (AC-30 and AC-30+12%CR) according to the Superpave Binder specifications using 1-) RTFOT and 2-) Modified Rotavapor aging processes. PG grades of these asphalt binders were not affected by using the Modified Rotavapor aging process in place of the RTFOT in the Superpave Binder Specifications.

CHAPTER 1
INTRODUCTION

1.1 Background

The Strategic Highway Research Program (SHRP) Superpave binder specifications require that an asphalt binder to be evaluated is to be subjected to a standard Rolling Thin Film Oven Test (ASTM D2872-97, 1999) (which simulates the effect of hot-mixing) to be followed by a Pressure Aging Vessel (PAV) process (which simulates several years of field aging). Study by the University of Florida and the FDOT has shown that, although this SHRP proposed aging method appears to be suitable for use on conventional asphalts, problems were encountered when modified asphalts were used. It was found that asphalts modified with coarse ground tire rubber (GTR) and SBR tended to spill out from the RTFOT bottles during the RTFOT process. When the TFOT process (ASTM D1754-97, 1999) was used in place of the RTFOT process, it was found that a thin skin tend to form on the surface of some modified asphalt samples, which reduced the homogeneity and the aging of samples. These problems with the RTFOT and TFOT processes on modified asphalts have also been reported by other researchers in the country.

GTR has been increasingly used in the friction course mixtures by the Florida Department of Transportation (FDOT) since 1988. A ten-year performance evaluation of asphalt-rubber friction course mixes in Florida has shown that the amount of cracking was relatively insignificant and rutting was less with the use of GTR (Choubane et al.,

1999). Due to the increasing use of GTR-modified asphalts and the potential use of other modified asphalts in Florida, it is crucial that a suitable laboratory aging method be developed to replace the RTFOT or the TFOT, so that the aging characteristics of these modified asphalts could be properly determined.

The rotavapor apparatus is commonly used for recovery of asphalts from solutions in accordance with ASTM D5404-97 (1999). In this standard test method, the solution of solvent and asphalt is distilled by partially immersing the rotating flask in a heated oil bath at a controlled temperature when solution is subjected to a partial vacuum and a flow of nitrogen or carbon dioxide. The rotavapor apparatus is also used to determine changes in mass and changes in the properties of asphalt due to heating in a German standard test (DIN 52016, 1988). In this application, the rotary evaporator is used without the condenser and receiver and the vacuum pump is replaced by an air pump in the test.

The rotavapor apparatus can be a more versatile aging device as compared with the thin film oven or rolling thin film oven. It can have the following advantages over these two conventional aging tests:

1. Different amounts of asphalt could be placed in the rotating flask for aging. Different sizes of rotating flask could be used to accommodate the different sample size. The possible use of a larger sample could eliminate the need to combine two or more samples to get enough materials to run the SHRP PAV or other tests.

2. There could be a better and more consistent heat transfer between the asphalt and the heated oil in the rotavapor apparatus due to the greater thermal mass of the oil as compared with the air in an oven.

3. Different aging effects could be achieved by using different combinations of temperature, flow rates of the injected air and rotating speed.

4. The flask rotation prevents skin formation on the asphalt.

5. The size of the flask and the tilting of the flask make spillage impossible.

1.2 Objectives of the Study

The main objectives of this research are as follows:

1-) To investigate the feasibility of the Rotavapor apparatus for simulating short and long-term aging of conventional and modified asphalts.

2-) To develop an effective and convenient test set-up and procedure which can be used in place of the RTFOT or the TFOT for simulation of aging effects of hot-mixing on conventional and modified asphalts.

3-) To develop an effective and convenient test set-up and procedure which can be used in place of the PAV procedure for simulation of long-term aging of conventional and modified asphalts in service.

4-) To evaluate the applicability of the new aging procedure to replace RTFOT and PAV processes in the SHRP binder specifications.

5-) To investigate the problem with extraction and recovery of rubber asphalt binder from rubber asphalt mixtures.

CHAPTER 2
LITERATURE REVIEW

2.1 Mechanism of Aging

2.1.1 Overview of aging mechanism

It is known that properties of asphalt change during the hot-mixing process while asphalt is in thin films and exposed to the surface of hot aggregates. This change which takes place during the hot-mixing process is called short-term aging. Typical changes in the properties during the hot-mixing process are decrease in penetration and increase in viscosity.

Asphalt cement continues hardening at a slower rate while it is stored in a silo, transported to pavement site, laid and compacted. After the pavement is compacted and opened to the traffic, asphalt cement also continues hardening very slowly during the service life of the pavement. The changes in the properties of asphalt cement which occur during the many years in the field is called long-term aging. The term "age hardening" is used for either short or long-term aging processes in the scientific literature.

Since asphalt is one of the main ingredients, the durability of asphalt effects the overall performance of an asphalt mix (Vallerga et al., 1957). One of the factors which effect the durability of asphalt is asphalt hardening. Asphalt hardening increases the stiffness and modulus of an asphalt concrete but lowers the strain required for cracking (Page et al., 1985). In other words, asphalt becomes brittle and easy to crack due to

hardening. As a result, the mechanism that causes the hardening of asphalt cement must be understood so the pavement engineers could design more durable pavements.

The factors which could affect the hardening of asphalts have been studied by many researchers. A total of six factors which contribute to the hardening of asphalt cements with time were explained by Vallerga et al. (1957). The factors listed include oxidation, volatilization, polymerization, thixotropy, syneresis, and separation. Clark (1956) studied the hardening effect of hot-plant mixing upon asphalts from many sources. He found that volatility of asphalt is a predominant factor in its eventual deterioration when used in a hot-mix pavement. Traxler (1963) stated a total of fifteen effects that may cause changes in the properties of asphalt cements. He explained nine additional factors over Vallerga et al. (1957). Effects that may reduce the adhesion of asphalts given by Traxler (1963) are shown in Table 2.1. Petersen (1984) indicated three fundamental composition-related factors that govern the changes that could cause hardening of asphalts in pavements as follows:

1. Loss of oily components by volatility or absorption by porous aggregates

2. Changes in the chemical composition of asphalt molecules by the reaction of asphalt with oxygen

3. Molecular structuring that produces thixotropic effects (steric hardening)

He concluded that the reaction of asphalt with atmospheric oxygen is a major factor leading to the hardening and embrittlement of asphalt. Shah (1978) found that asphalt cements with original high viscosity tend to harden more and at a rapid rate. He also indicated that there was no association between voids in the pavement and rate of hardening.

Table 2.1 Effects that may cause changes in the properties of asphalt cements.

Effects	Influenced by					Occurs		Ways to Retard
	Time	Heat	Oxygen	Sun Light	B&G rays	At Surface	In Mass	In general, Selected Source and Process
Oxidation	X	X	X	-	-	X	-	Inert atmosphere Free radical inhibitors
Photooxidation	X	X	X	X	-	X	-	Protection from light Inert atmosphere Free radical inhibitors
Volatilization	X	X	-	-	-	X	X	Protection from heat
Photooxidation reflected light	X	X	X	X	-	X	-	Protection from light Inert atmosphere Free radical inhibitors
Photochemical direct light	X	X	-	X	-	X	-	Protection from light Additives
Photochemical reflected light	X	X	-	X	-	X	X	Protection from light Additives
Polymerization	X	X	-	-	-	X	X	Free radical inhibitors
Thixotropy	X	-	-	-	-	X	X	Add dispersing agents Change source and processing of asphalt
Synerisis	X	X	-	-	-	X	-	Reduce paraffinic content
Changes by Nuclear Energy	X	X	-	-	X	X	X	-----
Action of Water	X	X	X	X	-	X	-	Change source and processing
Absorption by Solid	X	X	-	-	-	X	X	Improve dispersion of asphalt
Adsorption of components at solid surface	X	X	-	-	-	X	-	-----
Chemical reactions or catalytic effects at interface	X	X	-	-	-	X	X	-----
Microbiological Deterioration	X	X	X	-	-	X	X	Add fungiastic and bacteriostatic agents

Source:Traxler (1963)

However, when air void is considered at a selected age, Page et al. (1985) showed that recovered asphalts viscosity or penetration usually indicated harder asphalts for high air void content samples. They classified the variables which may contribute to the hardening of asphalts under four main categories. These are Plant Operations, Field Operations, Aggregates and Bitumen (Asphalt) characteristics. They noted that the most critical variable is probably the asphalt used in the hot mix. The effect of film thickness on age hardening was studied by Kandhal and Chakraborty (1996). It was found that accelerated aging would occur if the asphalt binder film thickness was less than 9 to 10 μm in an asphalt paving mixture compacted to 8 percent air void content. Recently, a new phenomenon called "physical hardening" has been observed in asphalt cements at low temperatures (Anderson and Marasteanu, 1999; Romero et al., 1999; Bahia and Anderson, 1993). Bahia and Anderson (1993) found that physical hardening is related to the nature of glass transition of asphalts and is mainly the result of isothermal volume shrinkage.

Since the asphalt composition varies depending upon the source of the crude oil, it is impossible to list all these factors which contribute to the hardening of asphalts in a strict order of importance (Traxler, 1961).

In conclusion, hardening of asphalt binder either during mixing or in service is an extremely complex phenomenon due to the numerous factors which affect it.

2.1.2 Oxidation

One of the most significant contributors to the hardening of asphalts is oxidation. It is the chemical reaction of asphalt with atmospheric oxygen. It is known that asphalt composition changes with time when the asphalt is exposed as a thin film to atmospheric

oxygen in the pavement. The hardening caused by oxidation is irreversible. The reaction rate depends on the character of asphalt and the temperature (Vallerga et al., 1957). According to Brock, the rate of chemical reaction between asphalt and oxygen will be twice as fast as at 300 °F than at 275 °F. The rate would also be four times faster at 325 °F than at 275 °F. Traxler (1963) showed that the rate of hardening, indicated by relative viscosity values, also increases with time. Since the asphalt is in thin films during mixing with hot aggregates, increase in mixing time will result in excessive hardening.

Brock stated that oxidation of asphalt is the concern of the Hot-mix Industry in four places.

2.1.2.1 Oxidation in the storage tanks

Oxidation occurs when the asphalt level drops below the heating coils. When coils are above the liquid level, they are covered with a thin film of asphalt. The thin film of asphalt now is exposed to hot surface of coils and in contact with air. Therefore, the oxygen in the air reacts with asphalt and oxidation occurs in the storage tanks. The oxidized asphalt on the coils is called "coke". To eliminate coking, asphalt level should not be allowed to drop below the heating coils.

2.1.2.2 Oxidation during mixing

During the mixing process, hot surface of aggregates is coated with thin asphalt films. Since the aggregates are continuously mixed, large surface area coated with thin films is exposed to oxygen in air. Therefore, asphalt will oxidize due to the reaction of asphalt with oxygen. The oxidation during mixing can not be totally eliminated. However, the rate of oxidation which takes place during the hot-mixing could be decreased by lowering the mixing temperature and time.

2.1.2.3 Oxidation of hot-mix on the road

Asphalt continues hardening at a slower rate even after compaction due to the oxygen entering into the pavement. In this case, the age hardening rate depends on the air void content and environment temperature (Tia et al., 1994). Using a dense surface mix will prevent oxygen penetration into the asphalt pavement. Therefore, progressive oxidation will be less in the pavement.

2.1.2.4 Oxidation in the hot-mix bins

Oxygen could be carried into the hot-mix bins in two ways

1. in the voids of the mix
2. through the cracks or openings in a bin wall.

Thus, asphalt will continue oxidizing in the hot-mix bins. Oxygen entering the bin in the voids of the mix can not be eliminated. However, the entrance of oxygen into the bin through the cracks could be eliminated by proper sealing.

The mechanism of oxidation is explained by Petersen (1984). He stated that components move from the more nonpolar to the more polar fractions, and strongly interacting, oxygen containing functional groups are formed during oxidation. These groups increase the viscosity of the asphalts. He concluded that ketones and sulfoxides are the major products formed during oxidation. Anhydrides and carboxylic acids are formed in smaller amounts as well.

2.1.3 Volatilization

Volatilization is the evaporation of the lighter constituents from asphalt, which is a mixture of hydrocarbons with a large range in molecular weight (Vallerga et al., 1957). Loss of the oily components of asphalt could be either by volatility or absorption by

porous aggregates. It takes place during the mixing in the hot-mix plant. Study made by Clark (1956) showed that the volatility of asphalt is a predominant factor in its eventual deterioration when used in hot-plant mixed pavement. However, Corbett and Merz (1975) found that the volatilization of light components was not significant with the binders tested in the Michigan Road test. Changes in the composition occurring in binder hardening were directionally the same in all cases. Napthene aromatics were converted to polar aromatics and then into asphaltenes. This indicates that the hardening was primarily caused by oxidation. Traxler (1963) stated that the importance of volatilization depends on the time and temperature to which asphalt is exposed and the nature and the viscosity of asphalt. He indicated that softer asphalts from a given source show more relative hardening caused by volatilization when the same time and temperature are used.

According to Petersen (1984), volatility loss is not a significant contributor to pavement hardening with current specifications and construction practices. Presently, there are two standard tests in the ASTM specifications to simulate aging which takes place during the hot-mixing in a plant operated at 150 °C. These are Thin Film Oven Test (TFOT) (ASTM D1754-97, 1999) and Rolling Thin Film Oven Test (RTFOT) (ASTM D2872-97, 1999). In both procedures, the weight loss is limited to 1 % to control the volatility loss in asphalt cements.

A variety of asphalts have been evaluated in different asphalt volatility tests by Zupanick and Baselice (1997). Data indicated that most of the asphalts in different volatility tests experienced mass loss less than 1 %. Also, some mass gain was observed

on a few asphalt samples. The mass gain in volatility tests could be explained by oxidation process and small amount of volatile loss.

Source of asphalt could also affect the rate of volatilization. Asphalts from heavy crudes (low API gravity) contain less light components compared to light crudes (high API gravity). Asphalts from heavy crudes are less likely to experience weight loss than those from light crudes will gain weight after the TFOT or RTFOT processes

In conclusion, the effect of volatilization on hardening of asphalts is not the major concern of today's researchers.

2.1.4 Physical hardening

Physical hardening has been observed in asphalt cements when they are exposed to low temperatures for long periods. As the temperature falls, asphalt shrinks in volume and hardening increases in asphalt. Shrinkage and hardening of asphalt continues even when the temperature stabilizes at a constant value (Background of SHRP Asphalt Binder Test Methods, 1993). Since asphalt hardening is a reversible process, the effects of physical hardening can be recovered by heating asphalt binders to room temperature. Since asphalt is highly sensitive to temperatures, it behaves differently at different temperatures. The behavior of asphalt from fluid to glassy occurs within a relatively narrow temperature range called the glass transition region. Bahia and Anderson (1993) found that physical hardening is related to the nature of glass transition of asphalts.

The Glass Transition Temperature (T_g) is measured by using different techniques. The investigation by Anderson and Marasteanu (1999) showed that T_g values obtained from dilatometry, differential scanning calometry and rheological considerations can be used interchangeably. The results indicated that physical hardening in asphalt binders

occurs above and below the glass transition temperature. For amorphous polymers, it occurs only below their glass transition temperature. Asphalts with higher wax content also showed stronger physical hardening effects both above and below their glass transition temperatures. In a study by Romero et al. (1999), the effect of physical hardening on low temperature properties of asphalt mixtures was studied. Study showed that factors such as mineral fillers and air voids of the mixtures had a greater influence than physical hardening on low temperature properties of mixtures.

2.2 Laboratory Aging Processes

2.2.1 Overview of laboratory aging

Asphalt aging process occurs in two stages. The first stage occurs during mixing in the hot-mix plant (short-term aging) and the second stage is in the field (long-term aging). Oxidation and loss of volatiles are two major mechanisms responsible from the changes in asphalt properties during the first stage. The second stage occurs in the field and is due to progressive oxidation. Air void content and atmospheric oxygen are the primary factors that affect the amount of progressive aging in the field.

Researchers have developed many methods which could simulate the aging characteristics of asphalt during mixing and service conditions. A review of the laboratory accelerated tests are given by Welborn (1984) and shown in Table 2.2. In the earlier methods, asphalts were aged in relatively thick films such as those used in TFOT and air blowing tests. In the early 1950's, some studies were made to develop testing apparatus to test asphalts in microfilm thicknesses (Welborn, 1984). However, the amount of aged residue was small in these tests.

Table 2.2. A review of laboratory accelerated tests.

Date	Investigator	Test Method	Evaluation Method
1903	Dow	30 hr, 400 °F	Changes in weight, penetration of residues
1903	Dow	Mixture aged for 30 min, 300 °F	Recovered asphalt, change in penetration
1937	Nicholson	Air blowing, 15 min., 425 °F	Penetration, ductility
1937	Rashig and Doyle	Air blowing, 15 min., 400 °F	Change in penetration
1937	Hubbard and Gollumb	Mixture, time and temperature varied	Recovered asphalt, change in penetration
1939	Lang and Thomas	Mixture, oven and outdoor aging	Change in mix properties, abrasion, strength, etc.
1940	Shattuck	Mixture, oven aging 30 min., 325 °F	Recovered asphalt, penetration, ductility, soft point
1940	Lewis and Welborn	1/8" film oven test, 5 hr., 325 °F (TFOT)	Change in weight, penetration, ductility
1946	Lewis and Halstead	1/8" film oven test, 5 hr., 325 °F (TFOT)	Change in weight, penetration, ductility
1952	Pauls and Welborn	Mixture, oven aging, TFOT	Compressive strength, residues
1955	Griffin et al.	Shell microfilm test, 5 μm, 2 hr., 225 °F	Viscosity before and after aging, aging index
1955	Heithaus and Johnson	Road tests, lab aging, microfilm	Recovered asphalts, microfilm aging index
1961	Traxler	TFOT and microfilm, 15 μm, 2 hr., 225 °F	Microviscosity at 77 °F compared
1961	Halstead and Zenewitz	TFOT and 15μm, 2 hr., 225 °F	Microviscosity at 77 °F compared
1963	Hveem et al.	Shell microfilm test, modified 20 μm, 24 hr., at 210 °F	Microviscosity at 77 °F before and after aging
1969	Hveem et al.	RTFOT and TFOT	Viscosity of RTFOT, TFOT, and recovered asphalts compared
1981	Schmidt and	RTFOT, 20 μm, 210 °F	Microviscosity of residue
1981	Kemp and Predoehl	Tilt Oven durability test, 168 hr., 235°F (California Tilt Oven)	Penetration, 77 °F ductility

Source: Welborn (1984).

Only two methods which simulated hot-mix plant aging successfully have been adopted as standard methods by ASTM and AASHTO. These are Thin Film Oven Test (TFOT) (ASTM D1754-97, 1999) and Rolling Thin Film Oven Test (RTFOT) (ASTM D2872-97, 1999).

The TFOT was developed by the Bureau of Public Roads in 1940 (Halstead and Welborn, 1974). In 1959, it was first adopted by AASHTO. ASTM accepted TFOT tentatively in 1960 and as a standard in 1969 (Halstead and Welborn, 1974). Rolling Thin Film Oven Test was first proposed in 1959 by the California Division of Highways. ASTM accepted RTFOT as a standard in 1970 (Halstead and Welborn, 1974). Both TFOT and RTFOT methods are intended to simulate aging which occurs during hot-mixing in a hot-mix plant operated at 150 °C.

2.2.2 Short-Term aging processes

Due to the common usage of TFOT and RTFOT by highway agencies, researchers have made extensive studies to evaluate these standard oven tests. Comparison between hot-mix and oven tests was made based on viscosity at 60 °C and 135 °C, penetration at 25 °C, infrared analysis and gel permeation chromatography by Jemison et al. (1991). It was found that the two oven tests, TFOT and RTFOT, do not accurately predict the chemical and physical changes occurring in the asphalts during the hot-mixing process. They concluded that the variables such as source of asphalts, presence of aggregates and incomplete extraction must be accounted while simulating hot-mix properties of asphalts.

Study by Shiau et al. (1992) has shown that it is possible to use TFOT and RTFOT at a higher temperature to simulate aging processes on asphalt binders in service.

TFOT and RTFOT tests were also evaluated by using conventional and modified asphalt binders by Phromsorn and Kennedy (1995). Results indicated that TFOT and RTFOT methods should not be used interchangeably for short-term aging. The study showed that the TFOT method produces less variability than the RTFOT method in most asphalt binders. They recommended to develop a new or modified short-term aging procedure.

Although TFOT and RTFOT aging methods seem to be suitable for use on conventional asphalts, problems were encountered when modified asphalts were used. Zupanick (1994) stated that a skin can form on the surface of modified asphalts and reduce the aging tendency. Bahia et al. (1998) indicated that modified asphalts because of their high viscosity do not roll inside the glass bottles and some tend to spill out from RTFOT bottles during the RTFOT process. Due to the shortcomings of TFOT and RTFOT processes with modified asphalts, alternative short-term aging procedures were studied. The RTFOT was modified by Bahia et al. (1998) to solve the problem of highly viscous asphalts. Modification was made by using steel rods inside the RTFOT bottles to create some shearing forces so the thin films inside the bottle could be spreaded uniformly. The results indicated that the use of rods changed the aging of filled asphalts significantly.

2.2.3 Long-Term aging processes

Asphalt continues hardening after being placed on the road due to progressive oxidation. The factors such as air voids, asphalt content and environment temperature also contribute to the hardening of asphalts in the field. Since aging is a continuous process, the long-term aging characteristics of asphalt cements need to be evaluated. The

most practical way of predicting the effect of aging within an asphalt binder is through laboratory simulation methods.

In a study by Petersen (1989), the Schmidt rolling microfilm test was modified to simulate field aging. In this thin film accelerated aging test (TFAAT), a 4.0 g sample was aged in a rolling thin film oven bottle to provide sufficient sample for chemical and rheological evaluation of the aged asphalt. It was found that properties of the aged asphalt is similar to those of field aged asphalt. Even though, the test simulated the field conditions fairly well, evaluation of the aged residue was limited to two tests due to small sample size.

Pressure Oxygen Bomb (POB) test was originally developed by Lee (1968). In this test, asphalt binders were subjected to pure oxygen under different levels at 150 °F to accelerate oxidative aging. Kim et al. (1987) modified the POB test to simulate oxidative aging that occurs after construction. Limited testing showed that asphalts subjected to pure oxygen at 100 psi pressure at 60 °C for 5 days had similar composition to those recovered from 5 to 10 years old field cores. POB test was also evaluated by Western Research Institute (WRI). The procedure developed for SHRP by WRI was a slight modification of that developed by Lee (1968). In the modified procedure, oxygen was replaced with air, the pressure of the reaction vessel was specified to be 2.07×10^6 pascals (300 psi) and the temperature was raised to 71 °C (Button et al., 1993). Since the degree of hardening caused by oxidation increases as aging temperature increases, SHRP raised the aging temperature and used a name called Pressure Aging Vessel (PAV) (SHRP Asphalt Binder Test Methods, 1993). The time for aging was decreased to 20 hours in the PAV process.

Some evidence has shown that the mechanism of oxidation at high temperatures is different than at low temperatures (Petersen, 1990). Thus, it would be appropriate to use low temperature to simulate long-term aging. Bell et al. (1991) also suggested to use a low pressure in the test due to the disruption of the sample when the gas pressure is relieved in the Pressure Oxidation test.

Different methods for short and long-term aging of asphalt-aggregate mixtures were also studied under SHRP. Two alternative procedures have been developed for long-term aging of compacted mixtures at Oregon State University. Long Term Oven Aging (LTOA) and Low Pressure Oxidation (LPO) tests were evaluated by Sosnovske et al. (1993). Mixtures were first subjected to short-term oven aging (STOA) by placing in a forced draft oven at 135 °C for a period of 4 hours with stirring once an hour to ensure that aging was uniform throughout the sample. For LTOA test, STOA aged specimens were compacted and placed in a forced draft oven preheated to 85 °C and left for 5 days. A temperature of 100 °C and a period of 2 days were also used as an alternative. Sosnovske et al. (1993) found that aging and testing of asphalt alone do not appear to be adequate for predicting mixture performance due to mitigating effects of aggregates on aging. To validate Short-Term Oven Aging (STOA) and Long-Term Oven Aging (LTOA) processes, samples were aged in STOA and LTOA procedures and compared to those obtained from different pavement sections by Bell et al. (1994). It was found that mixtures aged in the LTOA process at 85 °C for 2 days were representative of field pavements up to five years depending on the climate.

TFOT and RTFOT processes have been used at higher temperatures to simulate long-term field aging by Huang et al. (1996). Results showed that the RTFOT aging

process performed at a temperature of 185 °C for 75 minutes appear to simulate a 1 year field aging effect under typical Florida conditions. Bishara and McReynolds (1996) used a microwave oven with a frequency of 2.45 x 10^3 ±13 MHz and an output power of 1000 W to simulate both short and long-term aging characteristics of asphalts. Results indicated that TFOT+PAV and RTFOT+PAV aging was simulated in a microwave radiation for 158 minutes. Recently, a comparative study by Migliori and Corte (1998) showed that the asphalts aged in RTFOT+20 h. of PAV process were similar to those aged in 25 h. of PAV aging at 100 °C under 2.1 MPa (300 psi) pressure.

2.3 Rotavapor Apparatus

In order to determine the characteristics of asphalt in an asphalt paving mixture, it is necessary to extract the asphalt from the aggregate by means of a suitable solvent and then to recover the asphalt from the solvent without significantly changing the properties of asphalt (ASTM D5404-97, 1999). Rotavapor apparatus has been commonly used for the recovery of asphalts from solutions. This procedure has been standardized in ASTM D5404-97 (1999). In this standard test method, the solution of solvent and asphalt is distilled by partially immersing the rotating flask in a heated oil bath at a controlled temperature when solution is subjected to a partial vacuum and a flow of nitrogen or carbon dioxide.

The rotavapor apparatus is also used to determine changes in mass and changes in the properties of asphalt due to heating in a German standard test (DIN 52016, 1988). Rotary evaporator is used without condenser and receiver and the vacuum pump is replaced by an air pump in the test. A 100±1 g of asphalt sample is placed in a rotating flask, which is tilted at a 45° angle and fully immersed in a heated oil bath at 165±1 °C,

and rotated at 20±5 rpm. The sample is first heated up for 10 minutes. Then, air at ambient temperature at a rate of 500 ml/min is introduced into the flask and the sample is aged in the rotating flask for an additional 150 minutes. After the sample is aged, the mass loss of asphalt is determined.

2.4 Extraction and Recovery of Asphalt Binders

In order to study the properties of binder in the mix, asphalt binder is extracted from the mixtures and recovered from the solvents. Extraction methods to determine the asphalt cement content of the mixtures were developed during the period from 1900 to 1920 (Halstead and Welborn, 1974). Dow (1903), Bateman and Delp (1927), and Abson (1933) used different methods for the extraction and recovery of asphalt binders from solvents. ASTM tentatively accepted the procedures for extraction under the name called Quantitative Extraction of Bitumen from Bituminous Paving Mixtures(ASTM D2172-95, 1999) in 1963 and the method became standard in 1967 (Halstead and Welborn, 1974). For recovery, the Abson method was developed. Recovery of Asphalt from Solution by the Abson method (ASTM D1856-95a, 1999) was accepted by ASTM as a standard in 1961 (Halstead and Welborn, 1974). The rotavapor method for recovery of asphalt from solutions (ASTM D5404-97, 1999) was also accepted by ASTM as a standard in 1993.

The rotavapor is commonly used by the highway agencies including FDOT for recovery of asphalts. The common usage of the rotavapor in place of the Abson method was explained by Tia et al. (1994) that the vacuum in the rotavapor method enables a more complete separation of the solvent and reduces the effects of oxidation of asphalt.

Burr et al. (1990) also stated that the use of a vacuum during the initial recovery seems to reduce solvent hardening.

Research was conducted to evaluate statistical differences in extraction methods (ASTM D2172-95, 1999 Method A through E) by Stroup-Gardiner et al. (1989). Results of the investigation showed no statistical difference between either centrifuge, reflux or vacuum extraction methods, regardless of the type of solvent used for the test. However, the Abson Method (ASTM D1856-95a, 1999) requires that asphalt should be extracted from the aggregate-asphalt mixture in accordance with Method A of ASTM D2172-95 (1999) (centrifuge method) due to some evidence that recovered asphalt may have slightly lower penetration when extracted in the hot extraction methods.

To evaluate the effectiveness of recovery methods in removing solvents, Abson and rotavapor methods were studied by Burr et al. (1990). It was found that Abson method leaves some solvent to produce significant softening for large quantities of recovered materials and hardened asphalts obtained from aged pavement cores. Small amounts of solvent cause significant decreases in viscosity. Thus, it is necessary to remove solvent completely.

A variety of solvents have been used in the extraction of asphalt from solutions. The most common were trichloroethylene, 1,1,1-tricholoroethane, and methylene chloride. A number of solvents and mixtures with alcohols were compared with respect to their ability to remove the material by Cipione et al. (1991). Results indicated that trichloroethylene with 15 percent ethanol is the best solvent. Data also clearly indicated that no method removes all asphalt.

A report on extraction and recovery of asphalts published by AASHTO (1989) indicated that the coefficient of variance on recovered viscosities is 25 percent. Burr et al. (1991) stated that the error may be due to three main problems:

1. The solvent has some hardening effect on the asphalt that increases with temperature and time of exposure.

2. Incomplete removal of solvent from the asphalt during recovery.

3. Asphalt is not removed completely from the aggregate.

A study by Burr et al. (1991) showed that solvent aging appears to occur in all solvents. It could be minimized by using cold extraction and completing recovery of the resulting solutions as rapidly as possible.

Due to the problems with current extraction and recovery methods, Burr et al. (1993) designed a new apparatus and procedure for the purpose of extraction and recovery of asphalt binders from hot-mix and pavement samples for SHRP. Reproducibility tests indicated that the method is precise to about 7 percent for a standard deviation for a single operator. Tests also showed that little hardening occurs during the recovery for the SHRP method.

In the SHRP method, the apparatus consists of a rotary drum in which aggregate-binder sample is mixed with solvent. Then, the extract is filtered through both coarse and fine filters. Finally, the solvent is removed by using a rotavapor recovery procedure.

CHAPTER 3
DEVELOPMENT OF INITIAL MODIFIED ROTAVAPOR AGING APPARATUS

3.1 Preliminary Evaluation of the Rotavapor Aging Apparatus

A Rotavapor apparatus, which was originally used for recovery of asphalt from solvents after asphalt extraction, was modified to work as an aging device for asphalts and modified asphalts. The Rotavapor apparatus was modified such that the vacuum connection was replaced by an air pump with a controlled air flow. The original rotavapor apparatus is shown in Figure 3.1

The Rotavapor aging apparatus (as set up) was evaluated by running it on an AC-30 asphalt cement and a crumb rubber modified asphalt (AC-30 + 10% crumb rubber with a nominal size of #80 sieve). The CR-modified asphalt was made in the laboratory by blending the AC-30 with the CR at 170 °C using a high shear mixer. Two different sample sizes (50 & 75 g), two process temperatures (163 & 185 °C), two process durations (60 & 85 minutes), one air flow rate (4000 ml/min) and one rotating speed (60 rpm) were used in the Rotavapor aging process. The same asphalt and modified asphalt were also aged by the standard TFOT and RTFOT for comparison purpose. Figure 3.2 shows the rotavapor aging apparatus.

Penetration test at 25 °C and Brookfield viscosity test at 60 °C were performed on the original binders and on the aged binders after these various aging processes. One sample was used for each combination of test conditions. Penetration and viscosity test results are given in Appendix A.1 and A.2, respectively.

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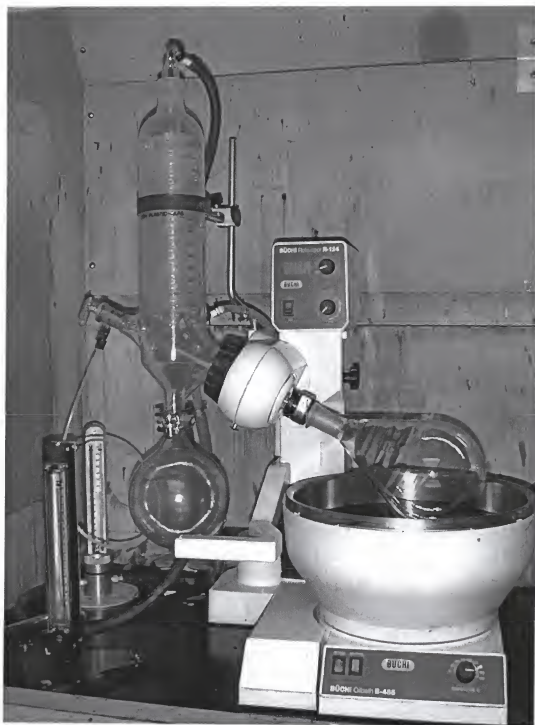
Figure 3.1. Original rotavapor apparatus used for recovery of asphalt from solvents.

Figure 3.2. Rotavapor aging apparatus.

Figure 3.3 shows the comparison of the effects of the standard TFOT and RTFOT on the penetration of the AC-30 asphalt & the CR-modified asphalt with those of the Rotavapor aging process (using 50 g samples and 85 min. duration) at 163 and 185 °C. The corresponding effects on the Brookfield viscosity at 60 °C are shown in Figure 3.4. It can be seen from Figures 3.3 and 3.4 that the aging severity of the Rotavapor process at 163 °C is slightly greater than that of the TFOT but slightly less than that of the RTFOT. However, the aging severity of the Rotavapor process at 185 °C is much greater than that of both the TFOT and RTFOT processes.

The effects of sample size, process duration and process temperature of the rotavapor process were evaluated. Figures 3.5 and 3.6 show the effects of these factors on the penetration at 25 °C and Brookfield viscosity at 60 °C, respectively, of the CR-modified asphalt which has been aged by the Rotavapor process. It can be clearly seen that sample size, process duration and process temperature all have significant effects on the aging severity. Smaller sample size, longer process duration, and higher process temperature produce significantly more severe aging effects.

3.2 Modified Rotavapor Aging Apparatus

Preliminary experimentation with this rotavapor aging apparatus showed that the oil bath was a major source of variation within this aging procedure and needed to be improved. The oil bath was exposed directly to the ambient air in the laboratory. When the oil bath was in use, it was losing heat to the environment through the side wall of the container as well as through the surface of the oil. Measurement of temperatures at different locations in the oil bath had shown a great variation of temperature within the oil bath. This condition of large temperature variation could result in the following major problems.

1. The asphalt binder in the rotating flask would not be heated evenly.

2. It would be extremely difficult to produce a consistent and repeatable aging effect on the asphalt binder in the rotating flask. The aging effect would be greatly influenced by factors such as (a) the height of the oil in the container, (b) the depth to which the rotating flask is placed within the oil bath, (c) the position of the thermal probe (used to control the heater) within the oil bath, and (d) the ambient air temperature.
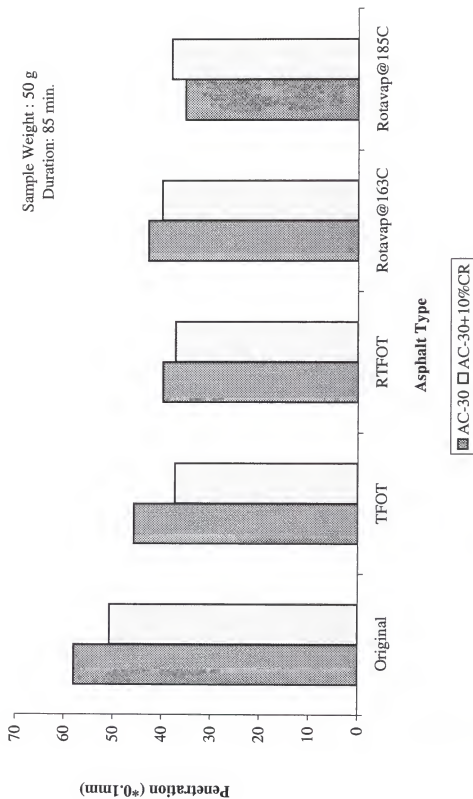
Figure 3.3. Comparison of penetration of asphalts after different aging processes.
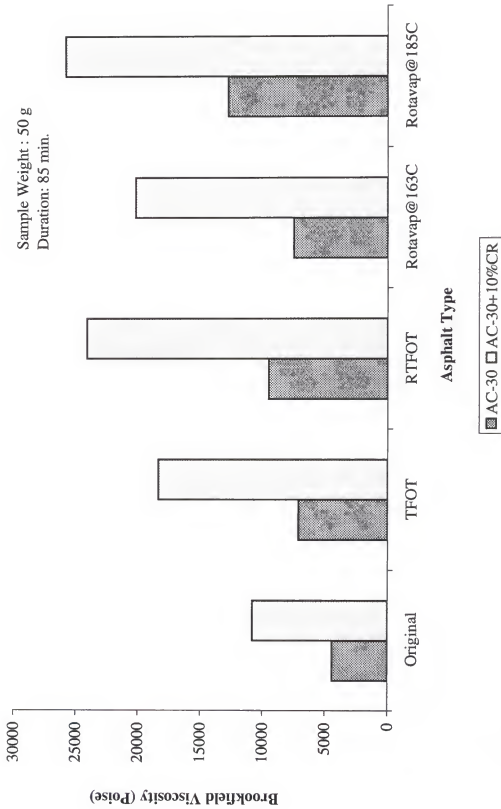
Figure 3.4. Comparison of Brookfield viscosity of asphalts after different aging processes.
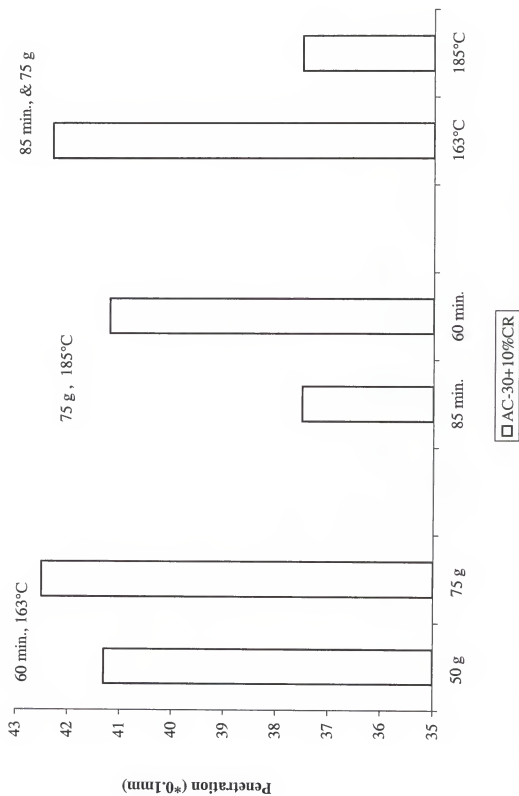
Figure 3.5. Effect of sample weight, aging duration and process temperature on penetration of crumb rubber asphalts.
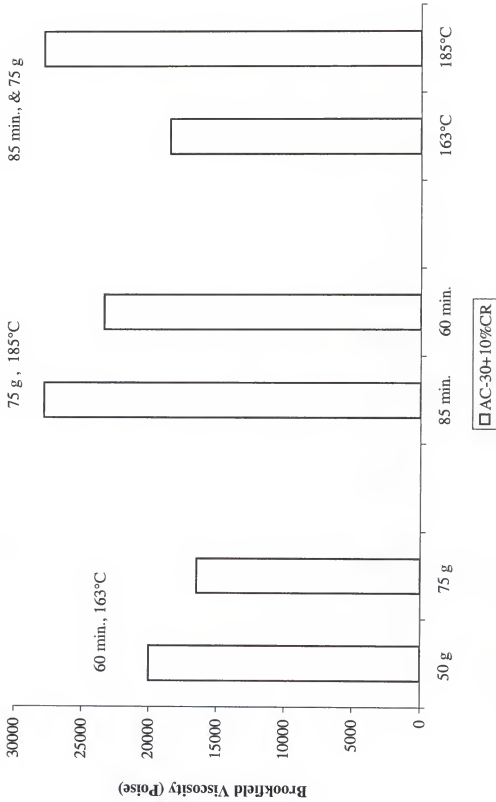
Figure 3.6. Effect of sample weight, aging duration and process temperature on Brookfield viscosity of crumb rubber asphalts.

In order to improve the uniformity of temperatures within the oil bath, modification of the oil bath for the modified rotavapor aging device was made. This was achieved by constructing a well-insulated box, and covering the oil bath with it.

Since the maximum heating temperature of the oil bath is about 175 C, the insulated box was designed to sustain a temperature up to 200 C. The outside wall of the box was made of a 24-gage galvanized steel sheet, while inside wall was made of a cold-rolled steel. A one-inch-thick insulating fiber material was placed between the galvanized steel sheet and the cold rolled steel. In order to be able to place the flask into the oil bath easily, a 229 X 254 mm (9 X 10 in.) door with a 102 X 102 mm (4 X 4 in.) glass window was placed on the front side of the insulated box. The purpose of having a glass window on the door is to enable a visual observation of the oil bath from the outside. The window material is a 0.25 in. thick tempered glass.

To determine the improvement in temperature uniformity within the oil bath, temperature measurements were made with a RTD thermal probe at different locations within the oil bath, with and without the insulated box covering. Figure 3.7 shows plots of temperature versus vertical distance from the surface of the oil, along the centerline of the cylindrical oil container, with and without the box covering. It can be seen that, for the oil bath without the box covering, the temperature varied from 177 °C at a depth of 8 cm to 142 °C at a depth of 2 cm. For the oil bath with the box covering, the temperature stayed fairly constant at 181 °C throughout the range of depth from 8 to 2 cm. For the case with the box covering, the temperature at the surface of the oil bath dropped only to 156 °C, while for the one without the box covering, it dropped drastically to 95 °C.

It could be seen from these data that the use of the insulated box covering can substantially improve the temperature uniformity in the oil bath.

The temperature variation in the horizontal direction at a constant depth in the oil bath was also investigated, with and without the box covering. Temperatures were measured with a RTD thermal probe at a constant depth of 8 cm, but at different horizontal positions within the oil bath. The temperature measurements are shown in Appendix A.3. Figure 3.8 shows plots of temperature versus horizontal distance from the center at a constant depth of 8 cm in the oil bath. It can be seen that, at this fixed depth, the temperature stayed fairly constant, with or without the box covering. For the case without the box covering, the temperature stayed constant at 177 °C. For the case with the covering, the temperature stayed at 181 °C. These two different oil temperatures were obtained by using the same setting on the temperature control of the rotavapor apparatus. Thus, it is clear that the settings for temperature control to produce the same oil temperature would be different for the two test set-ups.

In the adopted test set-up, the following settings of the Rotavapor apparatus were used:

1. The rotating flask is tilted at an angle of 45° and submerged in the oil bath as far down as possible, and such that the entire asphalt sample is below the oil level.

2. The rotating speed of the flask is set at 60 rpm.

3. The flow of air into the rotating flask is set at a rate of 4000 ml/min.

4. The thermal probe which measures and controls the temperature of the oil bath is placed at a depth of 8 cm from the surface of the oil and near the wall of the container.
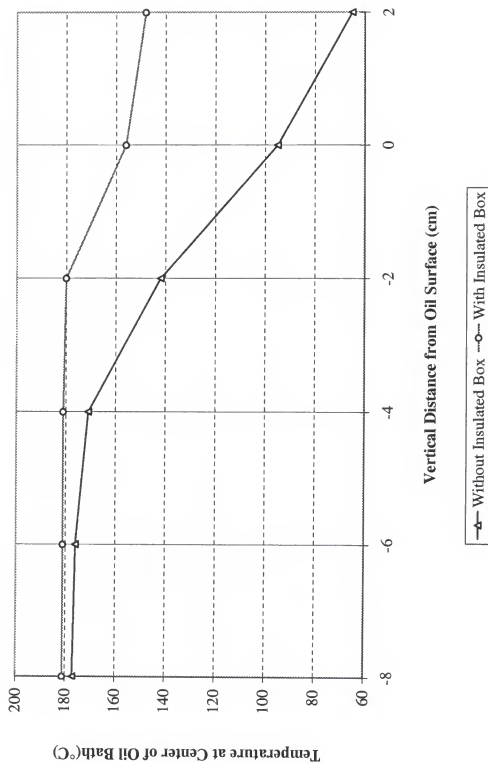
Figure 3.7. Plot of temperature versus vertical distance from surface of oil bath of modified rotavapor aging apparatus with and without insulated box.
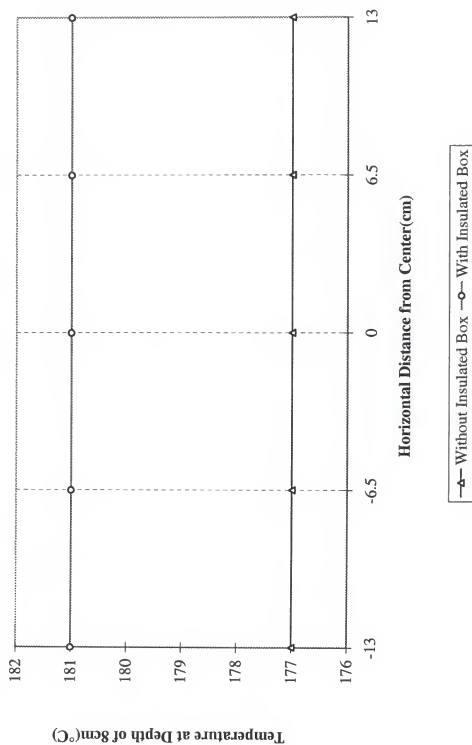
Figure 3.8. Plot of temperature versus horizontal distance from center at depth of 8 cm in oil bath of modified rotary aging apparatus with and without insulated box.

3.3 Testing Procedure Adopted

The adopted procedure for the modified Rotavapor aging process consists of the following main steps:

1. The oil bath is turned on at least one hour before the start of the test to ensure that the specified process temperature has been reached.

2. The asphalt sample to be tested is heated to give sufficient fluidity before it could be poured into the flask.

3. The flask is placed on a scale, and a specified amount of asphalt is poured into the flask.

4. The flask is then left to cool for at least an hour to room temperature before it is placed into the Rotavapor apparatus and submerged in the oil bath.

5. The window of the insulating box is closed immediately. The rotation of the flask and the air flow into the flask are turned on immediately to start the test.

3.4 Evaluation of the Modified Rotavapor Aging Apparatus

3.4.1 First factorial experiment

A full factorial experiment was conducted to evaluate the performance characteristics of the modified Rotavapor aging apparatus with the insulated covering. The schematic of the Modified Rotavapor Aging Apparatus is shown in Figure 3.9. The following variables were incorporated in the factorial experiment:

1. Two asphalt binders - AC-30 and AC-30 + 10%CR with a nominal size of #80 sieve

2. Three sample weights - 50, 125 and 200 g

3. Three process durations - 60, 85 and 110 minutes

4. One process temperature - 163 °C

Standard TFOT and RTFOT aging processes were also applied to the same binders to compare with the modified Rotavapor aging method. Two replicate samples were tested per combination of test parameters. After the asphalt binder had been aged in these processes, the following tests were run on each sample:

1. Standard penetration test at 25 °C (ASTM D5)

2. Brookfield viscosity test at 60 °C (ASTM D4402)

3.4.2 Test results and discussion of first factorial experiment

To observe the effects of process duration more easily, the average penetrations of the aged asphalt residues were plotted against process duration in Figure 3.10. It can be seen that the aging effect increases (as seen from the decrease in penetration) as process duration increases. Figure 3.11 shows the plots of the average penetrations of the aged residues versus sample weight. It can be seen that the aging effect decreases as the sample weight increases. The Brookfield viscosities of the residues were plotted against sample weight in Figure 3.12. It can be seen that the aging effect decreases (as seen from the decrease in viscosity) as the sample weight increases. Figure 3.13 shows the plots of the Brookfield viscosities of the residues versus process duration. The aging effect can be seen to increase as the process duration increases.
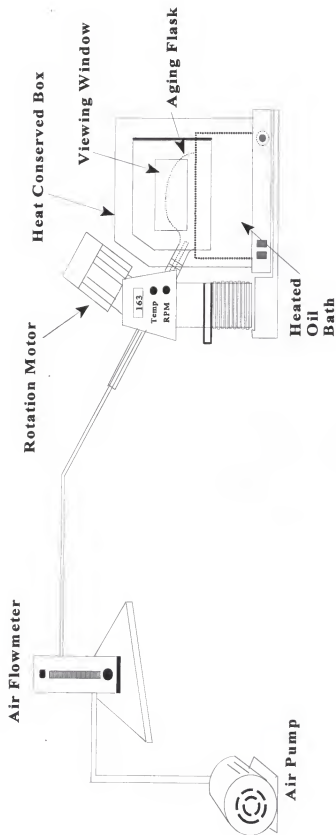
Figure 3.9. Schematic representation of modified rotavapor aging apparatus.
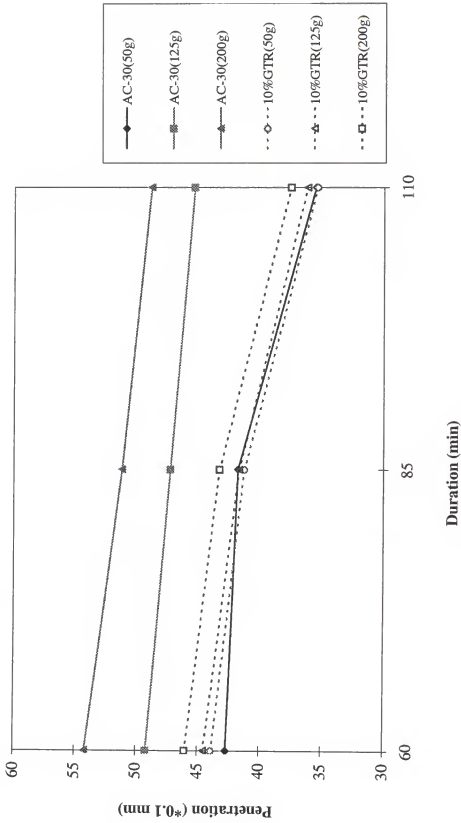
Figure 3.10. Plots of penetration of residues after modified rotavapor process at 163°C versus process duration.
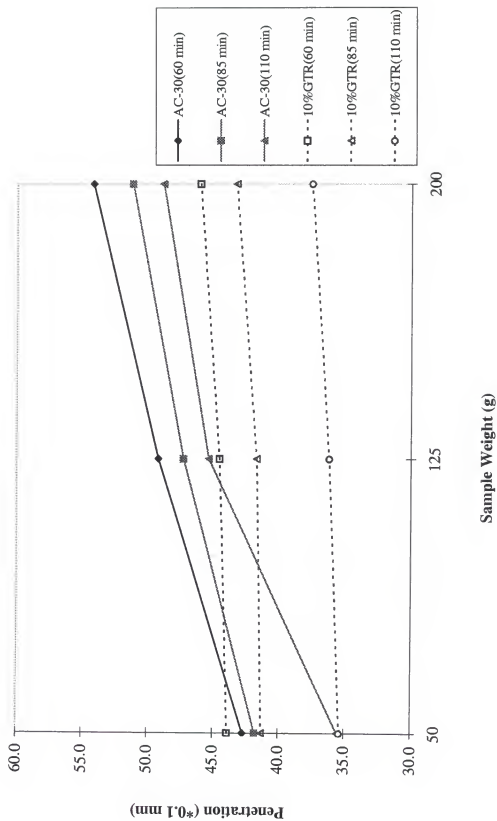
Figure 3.11. Plots of penetration of residues after modified rotavapor process at 163°C versus sample weight.

Figure 3.14 shows the comparison of the penetration of the aged residues after the various Rotavapor processes with those of the TFOT and RTFOT residues. It can be seen that the modified Rotavapor process at 163 °C for 60 minutes using a sample weight of 50 g is closest to the TFOT and RTFOT processes in aging severity. It is slightly more severe than the TFOT but slightly less severe than the RTFOT process. At this condition, the pure asphalt ages slightly more in the modified Rotavapor process as compared with the TFOT residue. However, at the condition, the crumb rubber modified asphalt ages slightly less in the modified Rotavapor process as compared with both the TFOT and RTFOT residues.

The original CR-modified asphalt was much harder(lower penetration) than the original pure asphalt. The aged CR-modified asphalt has a penetration very close to that of the aged pure asphalt. In the case of the insulated Rotavapor process for a duration of 60 minutes, the penetration of the aged CR-modified asphalt was even higher than that of the aged pure asphalt. This shows that the CR-modified asphalt generally ages less than the pure asphalt in terms of reduction in penetration. The comparison of the Brookfield viscosity of the aged residues after the various Rotavapor processes with those of the TFOT and RTFOT residues is shown in Figure 3.15. It can be seen that the comparison for the pure asphalts is different from that for the crumb rubber modified asphalts. For the pure asphalts, the modified Rotavapor process for 60 minutes using a sample weight of 50 g is closest to the TFOT, while the Rotavapor process for 85 minutes using a sample weight of 50 g is closest to the RTFOT in aging severity. For the crumb rubber modified asphalts, the modified Rotavapor process for 60 minutes using a sample weight of 50 g is closest to the TFOT, while the Rotavapor process for 110 minutes using a
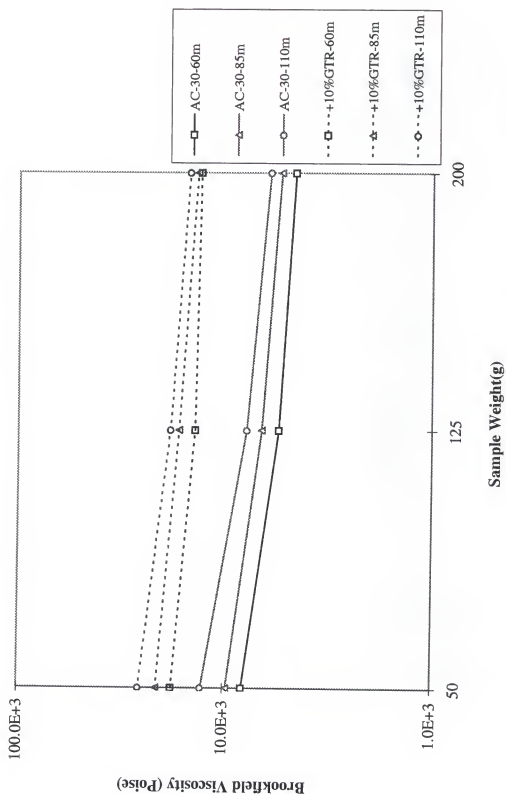
Figure 3.12. Plots of Brookfield viscosity of residues after modified rotavapor process at 163°C versus sample weight.
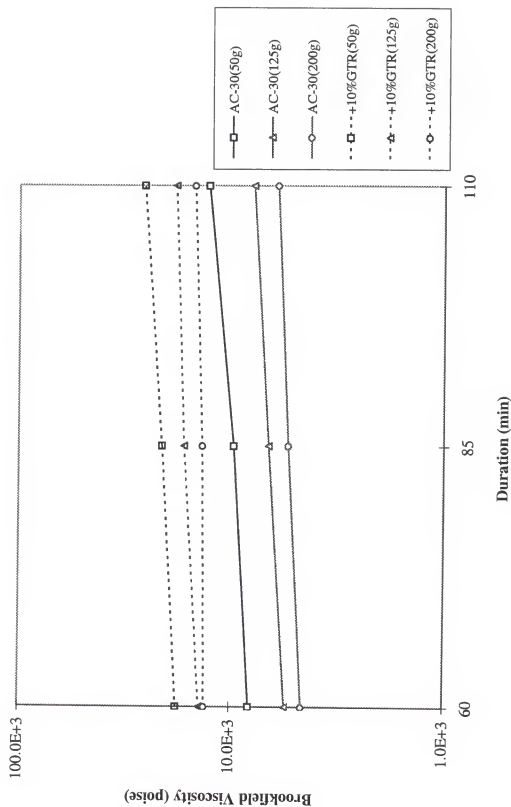
Figure 3.13. Plots of Brookfield viscosity of residues after modified rotavapor process at 163°C versus process duration.
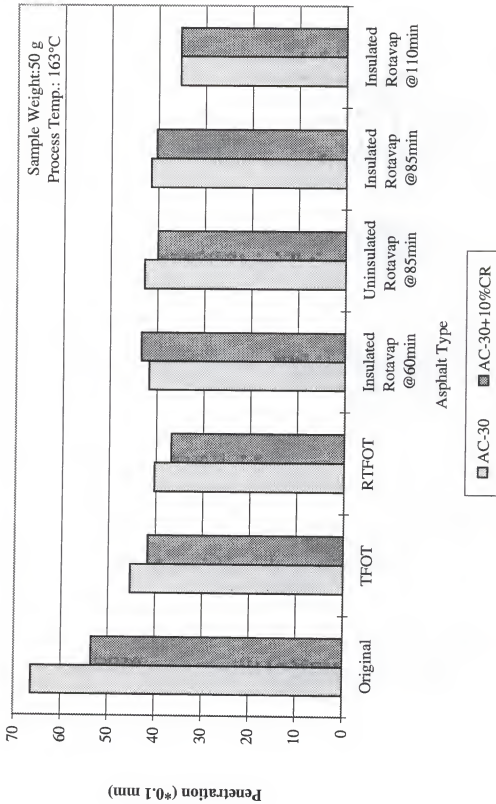
Figure 3.14. Comparison of penetration of aged residues.
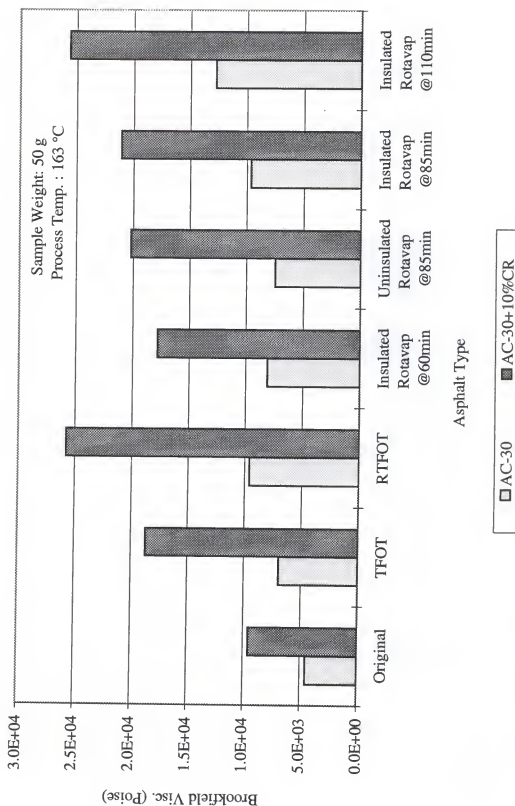
Figure 3.15. Comparison of Brookfield viscosity of aged residues.

sample weight of 50 g is closest to the RTFOT in aging severity. Penetration and viscosity test results are given in Appendix A.4 and A.5, respectively.

3.4.3 Second factorial experiment

To validate the results obtained from the first factorial experiment, additional crumb rubber asphalt samples from a different asphalt paving project were aged in the Modified Rotavapor aging process. The Modified Rotavapor aging apparatus with insulated covering is shown in Figure 3.16.

The following variables were incorporated in the factorial experiment.

1. One asphalt binder- AC-30+12 % crumb rubber with a nominal size of #80 sieve

2. Three sample weights- 50, 125, 200 g

3. Three process durations- 60, 85, and 110 minutes

4. Three process temperatures- 150, 163, 176°C

Samples were also aged in the standard TFOT and RTFOT aging processes to compare with the modified Rotavapor aging method. One sample was tested per combination of test parameters. The following tests were run on asphalt samples after they have been aged in these processes.

1. Standard penetration test at 25 °C (ASTM D5)

2. Brookfield viscosity test at 60 °C (ASTM D4402)

The results of penetration and viscosity tests are given in Appendix A.6 and A.7, respectively.
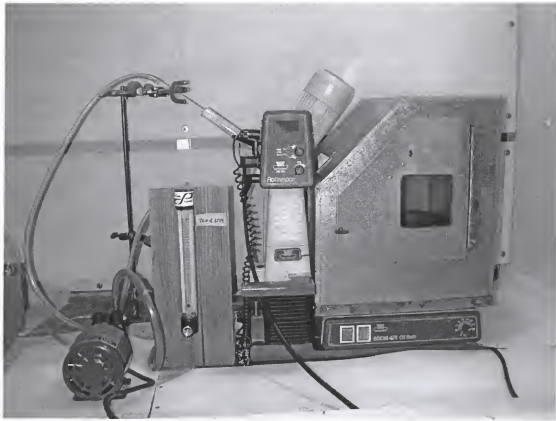
Figure 3.16. The Modified rotavapor aging apparatus with insulated covering.

3.4.4 Test results and discussion of second factorial experiment

The effects of process duration, sample weight and process temperature based on the penetrations of residues aged in the Modified Rotavapor process at 150 °C are shown in Figures 3.17, 3.18 and 3.19, respectively. Figure 3.20 shows the Brookfield viscosities of residues aged in the Modified Rotavapor process at 150 °C versus process duration. The effect of sample size based on the Brookfield viscosities of aged residues is shown in Figure 3.21. The plots of Brookfield viscosities of the aged residues versus process temperature are displayed in Figure 3.22.

It can be seen from Figures 3.17 through 3.22 that process duration, sample weight and process temperature all significantly affect the severity of aging. Longer

process duration, smaller sample size and higher process temperature increase the severity of aging.

Penetration of the crumb rubber asphalt residues aged in the Modified Rotavapor aging process is compared with those of TFOT and RTFOT residues in Figure 3.23. It can be seen that the Modified Rotavapor process at 163 °C for 85 minutes using a sample weight of 50 g is closest to TFOT, while the Modified Rotavapor process for 110 minutes is closest to RTFOT in aging severity.

The comparison of the Brookfield viscosities of aged residues with those of TFOT and RTFOT is shown in Figure 3.24. It can be seen that crumb rubber modified asphalt residues aged in the Modified Rotavapor process at 163 °C for 60 minutes using a sample weight of 50 g is closest to TFOT. Modified Rotavapor process for 110 minutes is closest to RTFOT in aging severity.
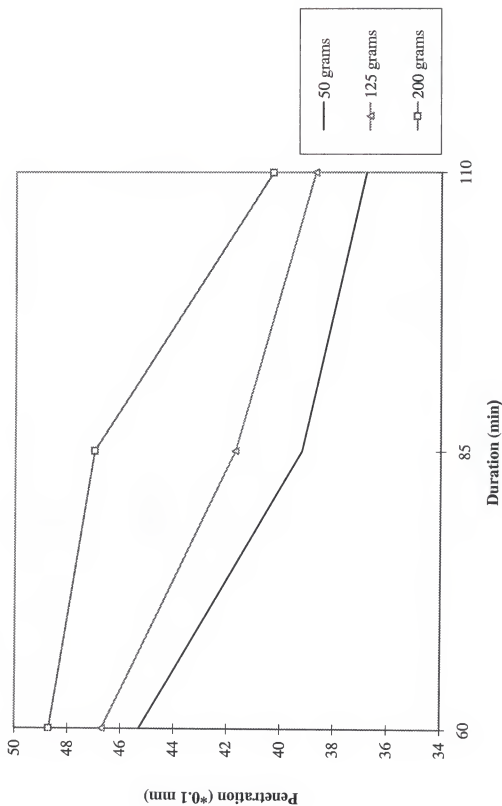
Figure 3.17. Plots of penetration of residues of AC-30+12%CR after modified rotavapor process at 150°C versus process duration .
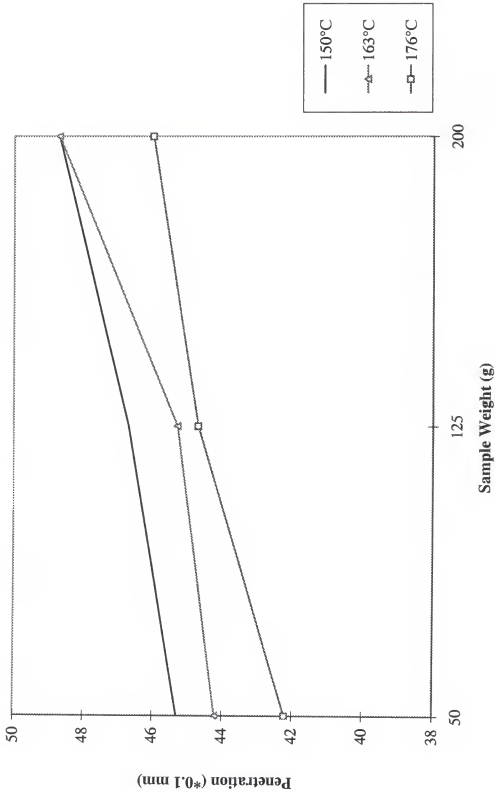
Figure 3.18. Plots of penetration of residues of AC-30+12%CR after modified rotavapor process for 60 min versus sample weight.
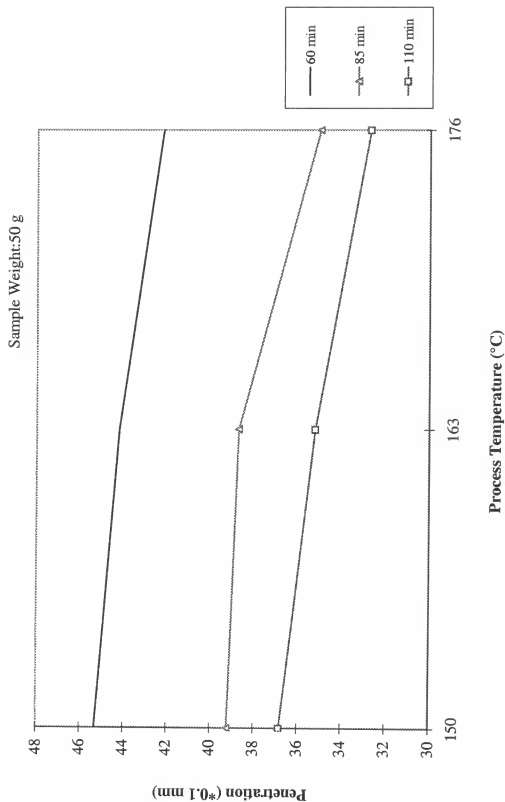
Figure 3.19. Plots of penetration of residues of AC-30+12%CR after modified rotavapor process versus process temperature.
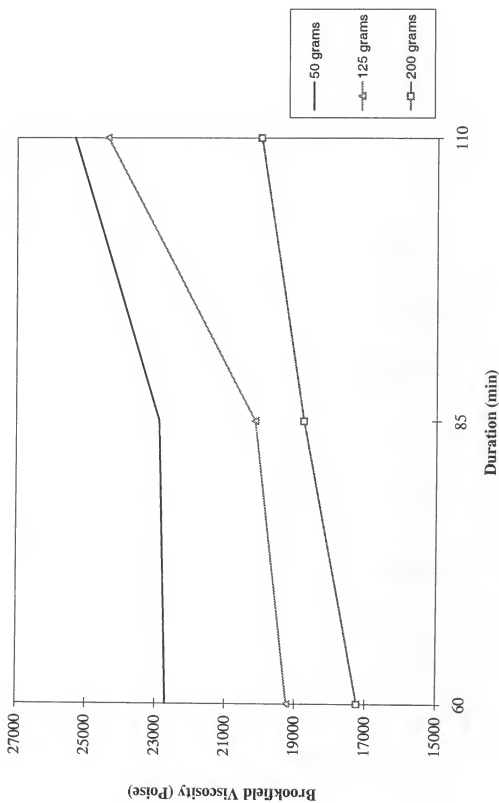
Figure 3.20. Plots of Brookfield viscosity of residues of AC-30+12%CR after modified rotavapor process at 150°C versus process duration.
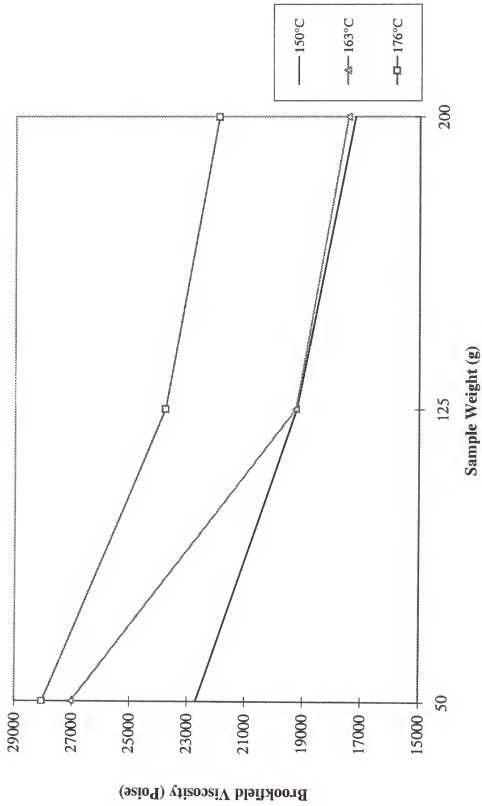
Figure 3.21. Plots of Brookfield viscosity of residues of AC-30+12%CR after modified rotavapor process for 60 min versus sample weight.
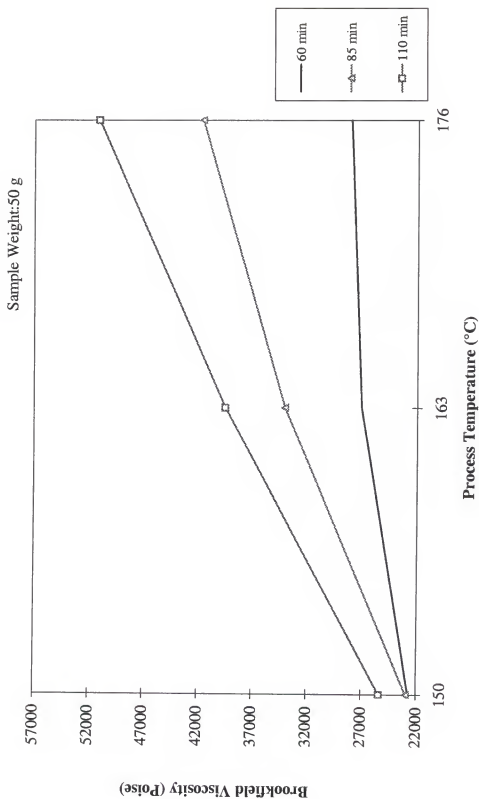
Figure 3.22. Plots of Brookfield viscosity of residues of AC-30+12%CR after modified rotavapor process versus process temperature.
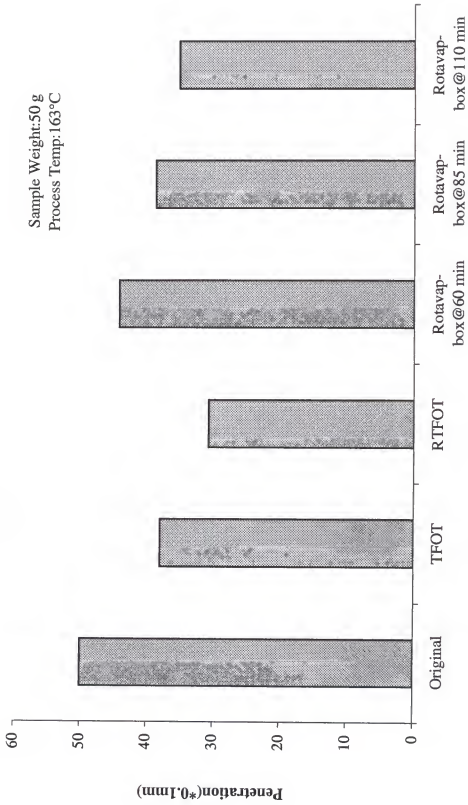
Figure 3.23. Comparison of penetration of asphalts after different aging processes.
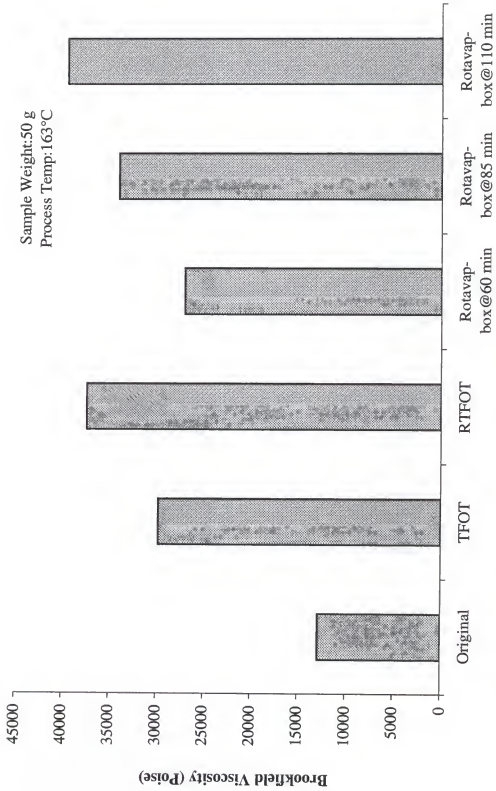
Figure 3.24. Comparison of Brookfield viscosity of asphalts after different aging processes.

3.5 Summary

The rotavapor apparatus used for recovery of asphalts was modified into an asphalt aging device as an alternative to the standard thin film oven and rolling thin film oven. To reduce the variation of aging condition due to variation of temperatures in the oil bath of the rotavapor apparatus, an insulated covering for the oil bath was constructed and used in this aging process. Evaluation results indicate that the aging severity of the modified rotavapor aging process is affected by variables such as process temperature, process duration and sample size. All these factors could be adjusted to produce the desired level of aging of asphalt samples. Because of the flexibility of controlling these variables in the modified rotavapor process, it appears that this method could be used to simulate the aging effects of the hot-mix plant process effectively.

CHAPTER 4
INVESTIGATION ON BINDER EXTRACTION FROM CONVENTIONAL AND CR-MODIFIED ASPHALT MIXTURES

4.1 Preliminary Evaluation of the Reflux Extraction Process

In order to calibrate the Modified Rotavapor Aging process, the properties of the rubber asphalts aged in the Modified Rotavapor process can be compared to the properties of the rubber asphalt binders recovered from the rubber asphalt mixtures.

To investigate the extraction of rubber asphalt binders from rubber asphalt mixtures, a total of four rubber asphalt mixture samples obtained from a SR-50 Paving project were tested by the Reflux Extraction method (ASTM D2172-95, 1999). 600-ml trichloroethylene (TCE) was used in the Reflux Extraction process. The mixture was a FDOT friction course (FC-2) mixture containing 6.34 % asphalt (AC-30)+ 0.76 % crumb rubber by the weight of the mix. The gradation of the aggregate used in the mix is given in Table 4.1.

Table 4.1. Gradation of aggregate for SR-50 mix.

SIEVE SIZE	% PASSING
½ "	100
3/8 "	97
No.4	34
No.10	12
No.40	11
No.80	6
No.200	4.5

The measured and computed results from reflux extraction tests on four crumb rubber asphalt mixture samples are shown in Table 4.2. It is assumed that the rubber asphalt mixtures contain 7.1 % asphalt+rubber and 92.9 % aggregate as specified in the design.

Table 4.2. Reflux extraction results for SR-50 mix.

		No. 1	no.2	No.3	no.4
A	Original Weight of Mix (g)	1244.1	1579.1	1371	1180.9
B=0.929 x A	Weight of Agg. in mix (g)	1155.8	1467	1273.7	1097
C	Extracted Weight of Agg.(g)	1158.9	1470.5	1284.1	1102.4
D	Amount of filler retained in bottles (g)	13.7	17.1	9.3	10.9
E= C+D	Total Extracted Weight of Agg. (g)	1172.6	1487.6	1293.4	1113.3
F= A-E	Weight of extracted binder(g)	71.5	91.5	77.6	67.6
G=F/A x100	% Binder extracted	5.75	5.79	5.66	5.72
H= E-B	Rubber+asphalt retained on filter paper and agg. (g)	16.8	20.6	19.7	16.3
I= H/A x100	% Rubber+asphalt retained on filter paper and agg. (g)	1.35	1.30	1.44	1.38

It can be seen from Table 4.2 that the computed values for % Binder extracted are much less than the design value (7.1 %). The difference is due to the asphalt+rubber which remain on the filter paper during the Reflux extraction process. From Table 4.2, The computed % rubber+asphalt retained on filter paper values for four samples were much greater than the % rubber (0.76 %). Thus, some of the asphalts also remained in the aggregates during the extraction process. This could be due to the absorptive nature of aggregates in Florida (limestone).

Two crumb rubber asphalt mixtures were also made in a laboratory at the University of Florida. The mixture was similar to the SR-50 mixture in aggregate gradation, rubber content and asphalt content. The gradation of aggregate used in the mix is shown in Table 4.3

Table 4.3. Gradation of aggregate used in the laboratory mix.

SIEVE SIZE	% PASSING
½ "	100
3/8 "	97
No.4	34
No.8	16
No.16	12
No.30	11
No.50	8.5
No.100	6
No.200	4.5

The reflux extraction results of two crumb rubber asphalt mixes are displayed in Table 4.4. It can be seen that the results of extraction tests on laboratory-produced mixtures are similar to those on the SR-50 mixture. The % extracted binder values were less that the design value (7.1 %). Also, the computed % binder+asphalt retained on the filter paper was greater than the % rubber in both cases.

Table 4.4. Reflux extraction results for laboratory rubber asphalt mixture.

		No. 1	No.2
A	Original Weight of Mix (g)	1210.9	1435.9
B=0.929 x A	Weight of Agg. in mix (g)	1125	1334
C	Extracted Weight of Agg.(g)	1117.2	1329.7
D	Amount of filler retained in bottles (g)	19.35	17.39
E= C+D	Total Extracted Weight of Agg. (g)	1136.6	1347.1
F= A-E	Weight of extracted binder(g)	74.3	88.8
G=F/A x100	% Binder extracted	6.14	6.18
H= E-B	Rubber+asphalt retained on filter paper and agg. (g)	11.6	13.1
I= H/A x100	% Rubber+asphalt retained on filter paper and agg. (g)	0.96	0.91

It is apparent that some of the asphalt also remained on the aggregate after the Reflux extraction process.

How much of the rubber and asphalt remain on the filter paper?. The investigation of this question is presented in the next section (4.2).

4.2 Determination of the Amounts of Rubber and Asphalt Which Could Not Be Extracted in the Reflux Extraction Process

During the preliminary evaluation of the extraction process on rubber asphalt mixtures, two major problems were encountered in the extraction of the rubber asphalt binder from the rubber asphalt mixtures.

1. It appears that most of the rubber remains on the filter paper in the Reflux extraction process.

2. A small amount of asphalt binder also remains in the aggregate and can not be completely extracted.

To solve these two problems, it was decided to determine the amounts of rubber and asphalt which could not be extracted, and add the right amounts of rubber and asphalt back to the extracted binder to form the corrected extracted binder from the mixture.

A total of six samples of a laboratory-produced rubber asphalt mixture were produced to be tested in the Reflux extraction process. The mixture was also similar to the SR-50 mixture in aggregate gradation, rubber content and asphalt content. The aggregate used was a Miami White Rock. The gradation of the aggregate used in the mix is shown in Table 4.5.

Table 4.5. Gradation of aggregate used in the laboratory mix.

Sieve Size	% Passing
½ "	100
3/8 "	97
No.4	34
No.8	16
No.16	12
No.30	11
No.50	8.5
No.100	6
No.200	4.5

The results of the Reflux extraction test on six rubber asphalt mixture samples are displayed in Table 4.6. It can be seen that the amount of binder+rubber which could not be extracted ranges from 0.75 % to 0.96 %. This is also greater that the total rubber % (0.76%) in the mix. This shows that some of the asphalt remains in the aggregates and could not be extracted completely.

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Table 4.6. Results of reflux extraction on rubber asphalt mixtures.

Sample No.	Original Weight (g)	Extracted Agg. Weight (g)	Binder Extracted (%)	Binder NOT Extracted (%)
1	1210.9	1136.55	6.14	0.96
2	1435.9	1347.09	6.18	0.92
3	1000	937.54	6.24	0.86
4	1000	936.82	6.31	0.79
5	1000	937.89	6.21	0.89
6	1000	936.45	6.35	0.75
Average			6.24	0.86
Standard Deviation (s)			0.079	0.079

To determine the amount of asphalt which is absorbed by the aggregates, six conventional laboratory-produced mixes were produced similar to the rubber asphalt mixture in aggregate gradation and asphalt content (6.34 %). The only difference was that the mixture had 0 % rubber. The results of the Reflux extraction test on six conventional mixture samples are given in Table 4.7.

Table 4.7. Results of reflux extraction on asphalt mixtures with no rubber.

Sample No.	Original Weight (g)	Extracted Agg. Weight (g)	Binder Extracted (%)	Binder NOT Extracted (%)
1	1000	939.09	6.09	0.25
2	1000	938.15	6.18	0.16
3	1000	940.18	5.98	0.36
4	1000	939.2	6.08	0.26
5	1000	940.55	5.95	0.39
6	1000	937.1	6.29	0.05
Average			6.095	0.245
Standard Deviation (s)			0.126	0.126

It can be seen from Table 4.7 that the amount of asphalt not extracted ranges from 0.05 % to 0.39 %, with an average of 0.25 % and a standard deviation of 0.126 %. Assuming that the same amount of asphalt also remained in the aggregates of the rubber asphalt mixtures, the average amount of rubber which was retained on the filter could be 0.86-0.25= 0.61 %, out of a total rubber content of 0.76 %. In other words, only about 20 % of the rubber in the rubber asphalt mixture was extracted during the Reflux extraction process.

4.3 Additional Reflux Extraction Tests

To validate the results of Reflux extraction tests in the previous section (4.2) and increase the number of data to determine the amounts of rubber and asphalt which remain on the filter paper, additional extraction tests were performed on both conventional and rubber asphalt mixtures. Ten samples of rubber asphalt mixtures and ten samples of conventional asphalt mixtures were produced in the laboratory and tested in the Reflux extraction process. The aggregate gradation and the binder contents of the mixtures were the same as those used in the SR-50 FC-2 mixtures. The rubber asphalt mixture contained 6.34 % AC and 0.76 % crumb rubber by the weight of the mix. The conventional asphalt mixture had 6.34 % asphalt by weight of the mix.

As the results were compared with those in Section 4.2, some inconsistencies were found. The investigation into the causes of the problem showed that the weights of the extracted aggregates were changing as they were left in the air. To investigate this problem, a series of tests were conducted on conventional and rubber asphalt mixture samples.

The weight of the extracted aggregate was measured at 0, 1, 2, 3, 6, 12 and 24 hours after it was dried in the oven. In order to investigate the possible effects of moisture, all samples were left to cool in a dessicator as well as in the air.

The weight of the extracted aggregate from conventional asphalt mix versus time is shown in Figure 4.1. It can be seen that the sample weight increases until 6 hours and than stabilizes. The increase in weight could be explained by the decrease in temperature and the absorption of moisture. The sample weight values in Figure 4.1 were also used to compute the AC content. Figure 4.2 shows the change in computed AC content versus time.

As mentioned earlier, extracted aggregate was also dried in the oven and left in a dessicator to cool. Figure 4.3 presents the sample weight of the extracted aggregate after drying in the oven and left in the dessicator versus time for a conventional mix. It can be seen that the weight increases until 3 hours and than stabilizes. The gain in weight in three hours is due to the decrease in temperature. The computed AC contents versus time is shown in Figure 4.4. Since the weight of extracted aggregate after 3 hours in a dessicator represents its true weight at a room temperature, this weight should be used for computing the extracted AC content. The computed asphalt contents based on the weights of the extracted aggregate left in a dessicator for 3 hours are given in Table 4.8. Sample weight and computed AC content results for conventional and rubber asphalt mixes are tabulated in Appendix A.8 and A.9, respectively.
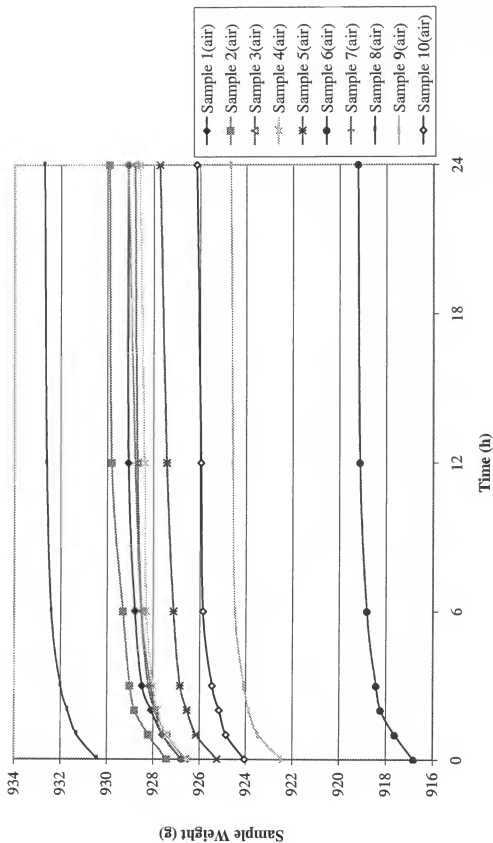
Figure 4.1. Sample weight after extraction vs time for conventional mixes left in air.
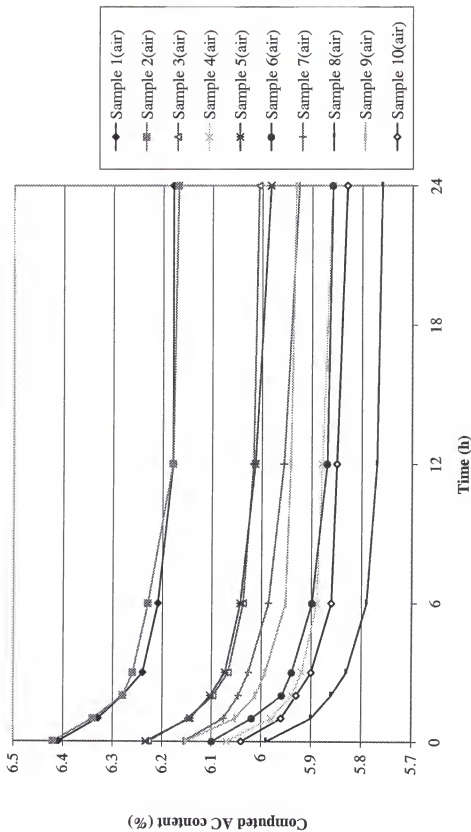
Figure 4.2. Computed AC content vs time for conventional mixes left in air.
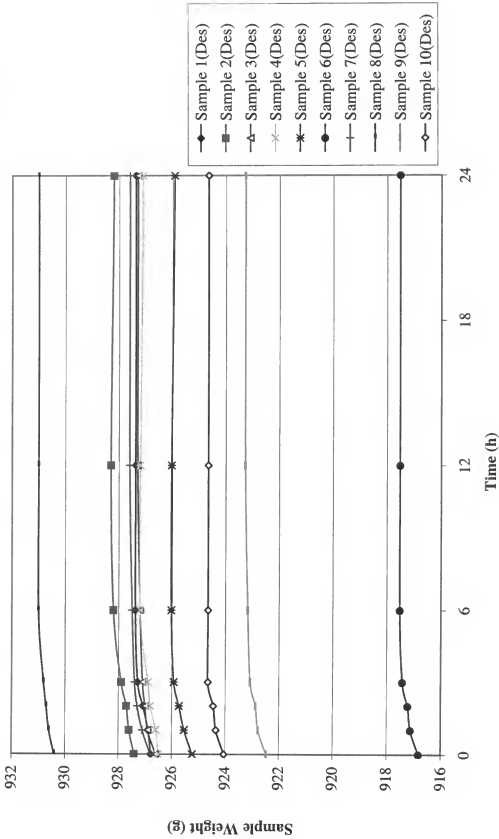
Figure 4.3. Sample weight after extraction vs time for conventional mixes left in dessicator.
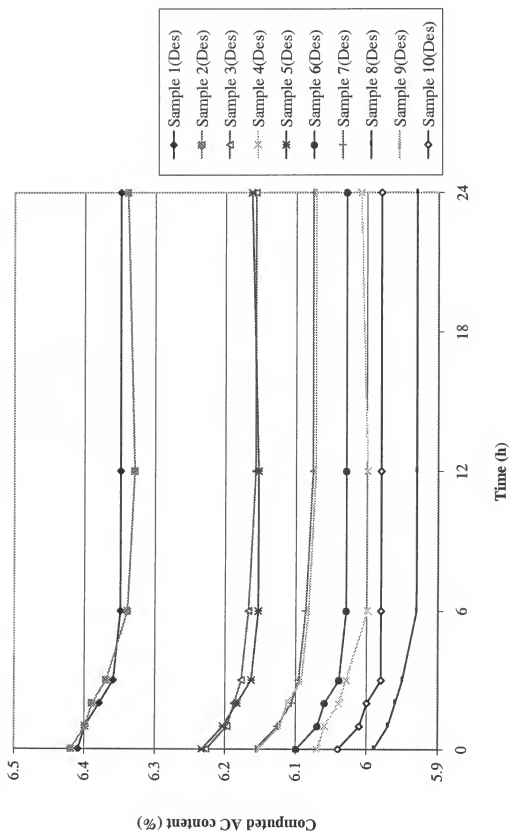
Figure 4.4. Computed AC content vs time for conventional mixes left in dessicator.

Table 4.8. Results of reflux extraction tests on conventional asphalt mixtures.

Sample No	Original Weight (g)	Extracted Agg. Weight (g)	Binder Extracted (%)	Binder Not Extracted (%)
1	1000	927.28	6.359	-0.019 *
2	1000	927.9	6.369	-0.029 *
3	1000	927.1	6.177	0.163
4	1000	926.9	6.029	0.311
5	1000	925.93	6.163	0.177
6	1000	917.42	6.039	0.301
7	1000	927.4	6.096	0.244
8	1000	930.8	5.95	0.39
9	1000	923.07	6.092	0.248
10	1000	924.65	5.98	0.36
Average			6.13	0.21
Standard Deviation (s)			0.144	0.144

* Negative values are due to the variation of test results

It can be seen that the average extracted asphalt content is 6.13 % for a mixture which had a 6.34 % of asphalt content and the average amount of asphalt which could not be extracted is 0.21 %.

The change in the weight of the extracted aggregate from the rubber asphalt mix after drying in the oven and left in air to cool with time is shown in Figure 4.5. It can be seen that the sample weight stabilizes after 6 hours. Figure 4.6 shows the computed extracted binder content as calculated from the weights of the extracted aggregate at different times. The weight of the extracted aggregate dried in the oven and left in a dessicator versus time for the rubber asphalt mix is presented in Figure 4.7. It can be seen that the weight increases within the first 3 hours, similar to the conventional mix. Figure 4.8 shows the computed extracted binder contents at different times for the rubber

asphalt mix. The computed extracted binder contents based on the weights of the
aggregate left in a dessicator for 3 hours are displayed in Table 4.9.

Table 4.9. Results of reflux extraction tests on rubber asphalt mixtures.

Sample No	Original Weight (g)	Extracted Agg. Weight (g)	Binder Extracted (%)	Binder Not Extracted (%)
1	1000	913.02	6.61	0.49
2	1000	910.54	6.693	0.407
3	1000	914.01	6.249	0.851
4	1000	916.23	6.408	0.692
5	1000	919.8	5.988	1.112
6	1000	914.01	6.448	0.652
7	1000	917.6	6.441	0.659
8	1000	920.86	6.205	0.895
9	1000	918.74	6.539	0.561
10	1000	922.0	6.15	0.95
Average			6.37	0.73
Standard Deviation (s)			0.22	0.22

It can be seen that the average extracted asphalt content is 6.37 % for a rubber
asphalt mixture which had a 7.10 % of asphalt content and the average amount of asphalt
which could not be extracted is 0.73 %. It can be assumed that the same amount of
asphalt (0.21 %) was retained in the aggregates for both conventional and rubber asphalt
mixes due to the same aggregate type, gradation and asphalt content used in both mixes.
The average amount of rubber which can not be extracted is 0.73-0.21= 0.52 %. Thus,
the total amount of rubber not extracted in the Reflux process will be
(0.52/0.76)x100%=68%.
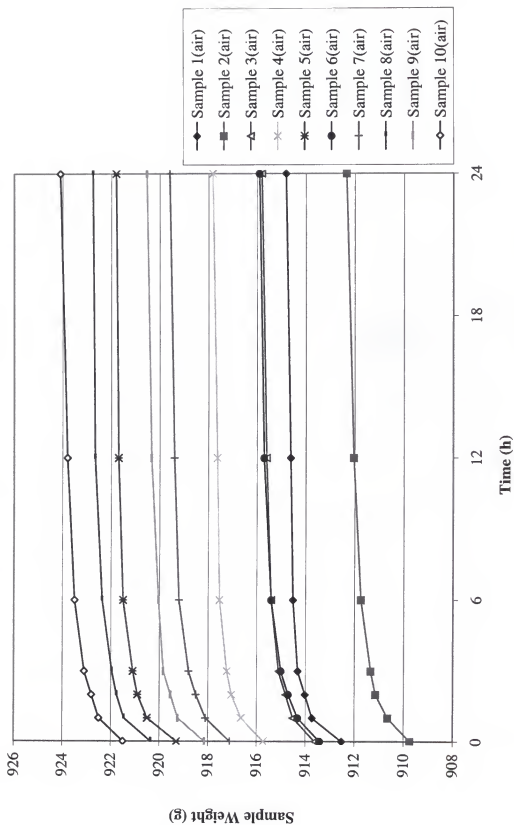
Figure 4.5. Sample weight after extraction for rubber asphalt mixes left in air.
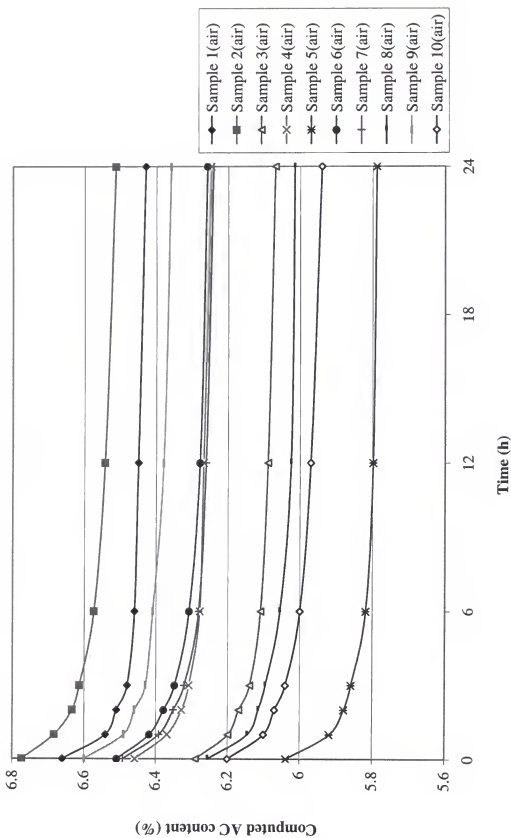
Figure 4.6. Computed AC content vs time for rubber asphalt mixes left in air.
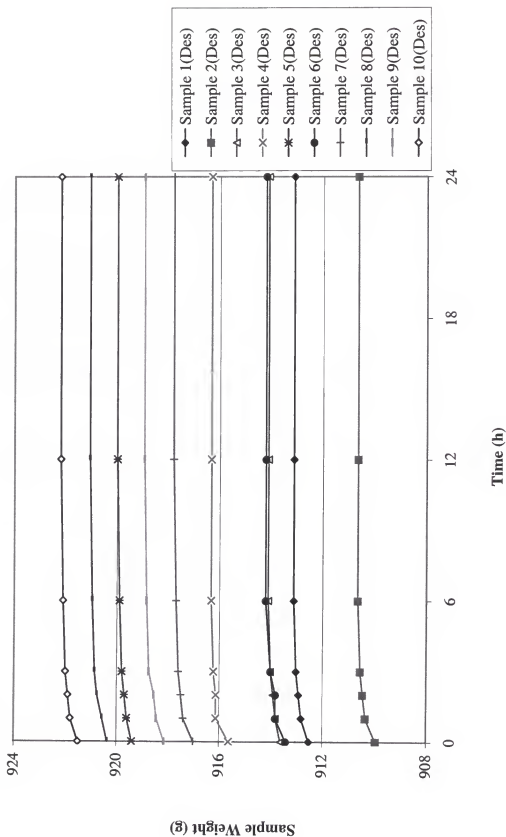
Figure 4.7. Sample weight after extraction vs time for rubber asphalt mixes left in dessicator.
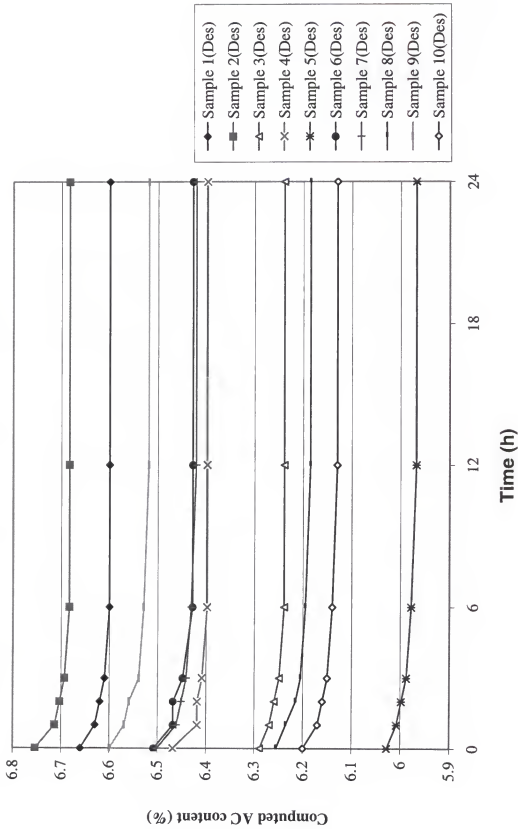
Figure 4.8. Computed AC content vs time for rubber asphalt mixes left in dessicator.

4.4 Summary

In order to compare the properties of the rubber asphalts aged in the Modified Rotavapor process with those recovered from the rubber asphalt mixtures, Reflux extraction tests were run on both conventional and CR-modified asphalt mixes. Based on our investigation, some of the findings are as follows:

1. Most of the rubber remains on the filter paper in Reflux extraction process and can not be completely extracted.

2. A small amount of asphalt binder also remains on the aggregate and can not be completely extracted

3. The amount of rubber and asphalt which remain on the filter paper varies from mixture to mixture.

4. Since the weight of extracted aggregate after 3 hours in a dessicator represents its true weight at a room temperature, this weight should be used for computing the extracted AC content

CHAPTER 5
REFINEMENT OF THE MODIFIED ROTAVAPOR AGING METHOD

5.1 Refinement by using a Morton Flask

In order to improve the effectiveness of the Modified Rotavapor aging method, which was discussed in detail in Chapter 3, the set-up was refined by replacing the standard distillation flask with a Morton flask. The Morton flask with its indented shape has two advantages over the standard distillation flask as follows:

1. It reduces the temperature variation in the oil bath by stirring the oil more vigorously

2. It provides better agitation of thin films inside the rotating flask

The standard distillation and Morton flasks are shown in Figure 5.1. To evaluate the effects of the Morton flask on the temperature variation within the oil bath, a RTD probe was used to measure temperatures at different locations in the oil bath when these two different flasks were used. Figure 5.2 shows the plot of temperature versus vertical distance from the surface of the oil bath for both conditions. It can be seen that the temperature in the oil bath varied from 175 to 151 °C when the Morton flask was used. However, the temperature varied from 175 to 144 °C when the standard distillation flask was used. This clearly indicates that the use of Morton flask reduces the temperature variation in the oil bath. Since the inside of the Morton flask is rougher than that of the standard distillation flask, it's believed that thin asphalt films will be agitated better in the Morton flask. The temperature measurements are given in Appendix A.10.

Conventional Flask   Morton Flask

Figure 5.1. Conventional and Morton flasks.
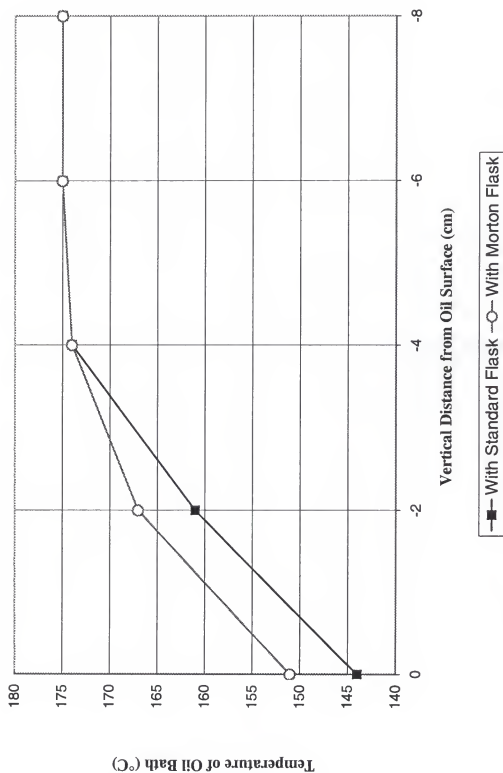
Figure 5.2. Plot of temperature versus vertical distance from surface of oil bath of the modified rotavapor aging apparatus with standard and morton flasks.

5.2 Preliminary Evaluation of the Improved Rotavapor Aging Apparatus with Morton Flask

5.2.1 Preliminary factorial experiment

A parametric experimental study was conducted to evaluate the characteristics of the Improved Rotavapor aging apparatus with the Morton flask. The following test variables were used in the parametric study:

1. Three sample weights- 125, 200 and 275 g

2. Two process temperatures- 163 and 176 °C

3. Eleven process durations- 85, 110, 135, 160, 185, 210, 260, 285, 335, 435 and 615 minutes

4. Rotating Speed of the Morton flask- 20 rpm

5. Air Flow- 4000 ml/min

A crumb-rubber modified asphalt binder (AC-30+12%CR) from a SR-580 paving project was tested for the preliminary evaluation. Three samples per condition were tested for the process temperature of 163 °C. Only one sample per condition was tested while using a process temperature of 176 °C. The same CR-modified asphalt binder was also subjected to the following standard aging procedures:

1. TFOT

2. RTFOT

3. TFOT+PAV @100 °C

4. RTFOT+PAV @100 °C

After the CR-modified asphalt binders were aged in the different aging processes, all of the aged residues were characterized by means of the following tests:

1. Standard Penetration Test at 25 °C

2. Brookfield Viscosity Test at 60 °C

3. Dynamic Shear Rheometer Test at 25 and 64 °C

4. Mass change determination

5.2.2 Test results and discussion of the preliminary factorial experiment

The asphalt binders which were aged in the Modified Rotavapor aging apparatus using different testing configurations were compared with those aged in the standard aging processes. The comparison was made to determine the test configuration for the Modified Rotavapor aging procedure that would produce similar aging effects and severity as the standard processes. Penetration at 25 °C, viscosity at 60 °C, G* at 25 °C, delta at 25 °C, G* at 64 °C and delta at 64 °C of AC-30+12%CR residues after the Modified rotavapor aging process are given in Appendices A.11 through A.16, respectively. The same binder test results of AC-30+12%CR residues after different aging processes are shown in Appendix A.17.

Based on the penetration at 25 °C of the aged residue, the Modified Rotavapor process at 163 °C for 160 minutes and using a sample weight of 200 g is closest to TFOT, while the Modified Rotavapor process at the test configuration of 163 °C, 210 minutes and 200 g is closest to RTFOT. The Modified Rotavapor process at the test configuration of 163 °C, 615 minutes and 125 g is closest in aging severity to TFOT+PAV and RTFOT+PAV. The comparison of the penetration of the aged residues under these various aging conditions can be seen in Figure 5.3.

On the basis of Brookfield viscosity at 60 °C, the Modified Rotavapor process at 163 °C, 160 minutes and 200 g is closest to TFOT, while the Modified Rotavapor process

at 163 °C, 210 minutes and 200 g is closest to RTFOT. The Modified Rotavapor process at 163 °C, 615 minutes and 125 g is closest to TFOT+PAV and RTFOT+PAV. Figure 5.4 shows the comparison of the Brookfield viscosity of aged residues under these aging conditions.

Based on the G* at 25 °C, the Modified Rotavapor process at 163 °C, 160 minutes and 200 g is closest to TFOT, while the Modified Rotavapor process at 163 °C, 210 minutes and 200 g is closest to RTFOT. The Modified Rotavapor process at 163 °C, 615 minutes and 125 g is closest to TFOT+PAV. The comparison of the G* values at 25 °C of the aged residues under these aging conditions are shown in Figure 5.5.

On the basis of δ at 25 °C, the Modified Rotavapor process at 163 °C, 185 minutes and 200 g is closest to TFOT, while the Modified Rotavapor process at 163 °C, 210 minutes and 200 g is closest to RTFOT. The Modified Rotavapor process at 163 °C, 615 minutes and 125 g is closest to TFOT+PAV. Figure 5.6 shows the comparison of δ values at 25 °C of the aged residues under these aging conditions.

Based on the G* at 64 °C, the Modified Rotavapor process at the test configuration of 163 °C, 185 minutes and 200 g is closest to TFOT and RTFOT. The Modified Rotavapor process at 163 °C, 615 minutes and 125 g is closest to TFOT+PAV. The comparison of the G* values at 64 °C of the aged residues under these aging conditions are displayed in Figure 5.7.

On the basis of δ at 64 °C, the Modified Rotavapor process at 163 °C, 135 minutes and 200 g is closest to TFOT, while the Modified Rotavapor process at 163 °C, 210 minutes and 200 g is closest to RTFOT. The Modified Rotavapor process at 163 °C, 615 minutes and 125 g is closest to TFOT+PAV and RTFOT+PAV.
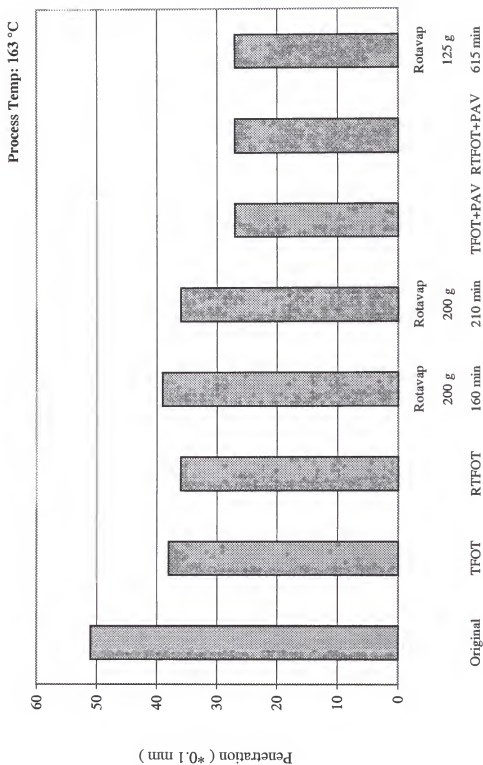
Figure 5.3. Comparison of penetration of asphalts after different aging processes.
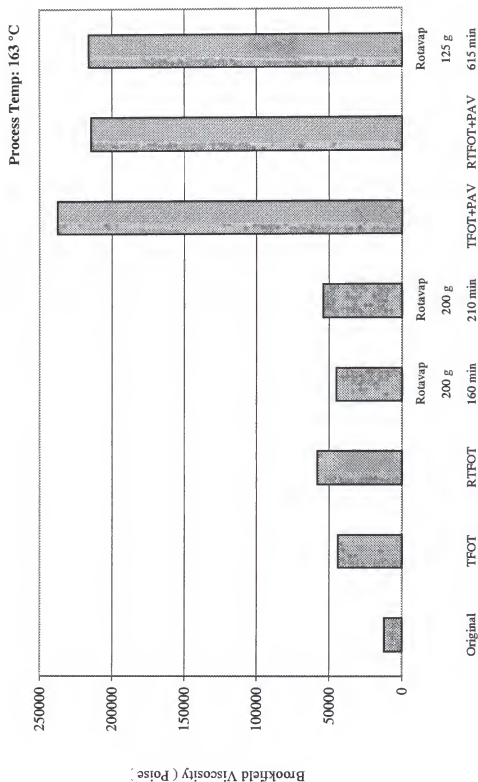
Figure 5.4. Comparison of Brookfield viscosity at 60 °C of asphalts after different aging processes.
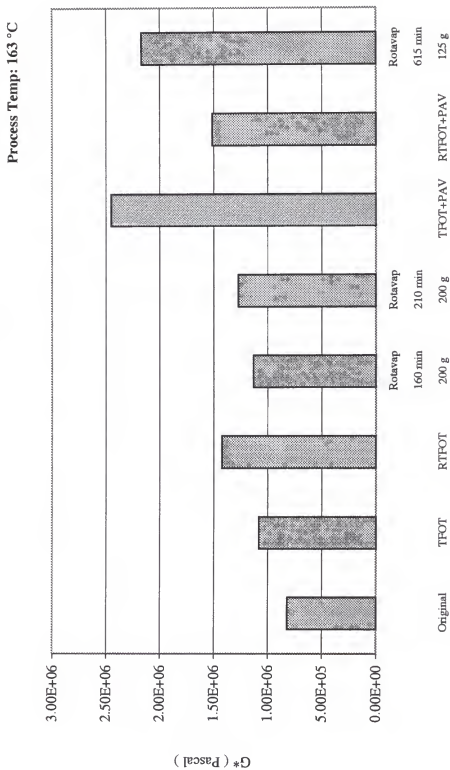
Figure 5.5. Comparison of dynamic modulus (G*) of asphalts at 10 rad/sec and 25 °C after different aging processes.
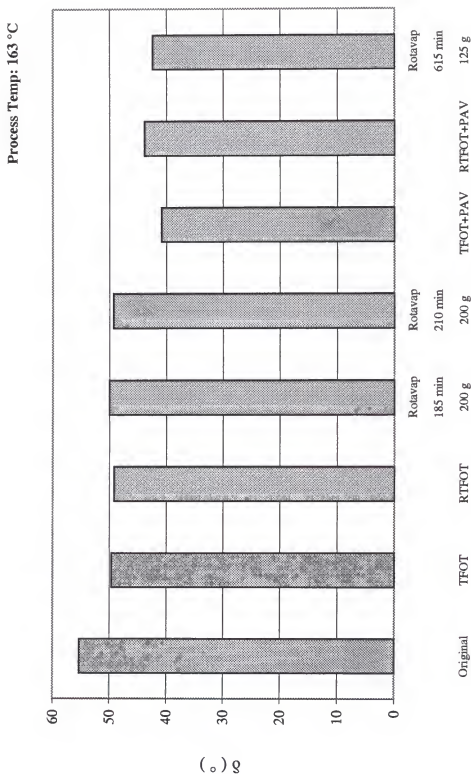
Figure 5.6. Comparison of phase angle (delta) of asphalts at 10 rad/sec and 25 °C after different aging processes.
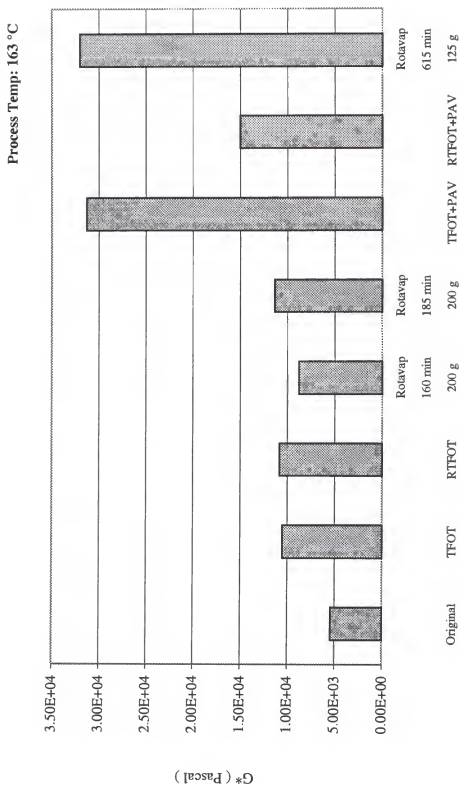
Figure 5.7. Comparison of dynamic modulus (G*) of asphalts at 10 rad/sec and 64 °C after different aging processes.
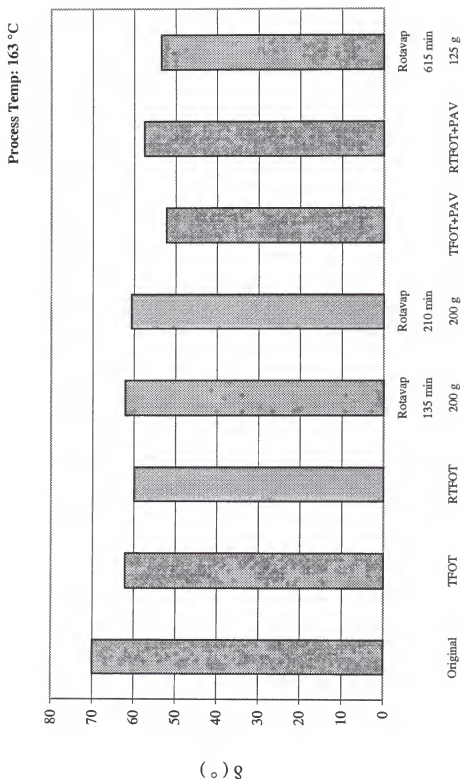
Figure 5.8. Comparison of phase angle (delta) of asphalts at 10 rad/sec and 64 °C after different aging processes.

Figure 5.8 shows the comparison of δ values at 64 °C of the aged residues under these aging conditions. Based on the penetration, viscosity and DSR test results of aged residues, the three testing configurations of the Modified Rotavapor process which produce aging severity closest to TFOT and RTFOT are as follows:

1. 163 °C, 200 g and 160 minutes

2. 163 °C, 200 g and 185 minutes

3. 163 °C, 200 g and 210 minutes

Comparison of the measured mass change of the samples after aging in the TFOT, RTFOT and these three testing configurations of the Modified Rotavapor process is shown in Figure 5.9. It can be seen that the the mass loss of the binders in the Modified Rotavapor aging process is very close to those in TFOT and RTFOT.

It appears that a sample weight of 125 g, 163 °C and 615 minutes in the Modified Rotavapor aging process produce the aging severity closest to TFOT+PAV and RTFOT+PAV. Since the temperature used in this test configuration is much higher than the actual field temperature, it may not be appropriate to simulate the long-term field aging characteristics of asphalt binders using this temperature (163 °C) in the Modified Rotavapor aging process. If a lower temperature is used, it may be more realistic. However, when the lower temperature is used in the Modified Rotavapor aging process, asphalts modified with polymers may not roll inside the rotating flask due to their high viscosity. Thus, a higher temperature is needed to process these modified asphalts in the Modified Rotavapor aging process.
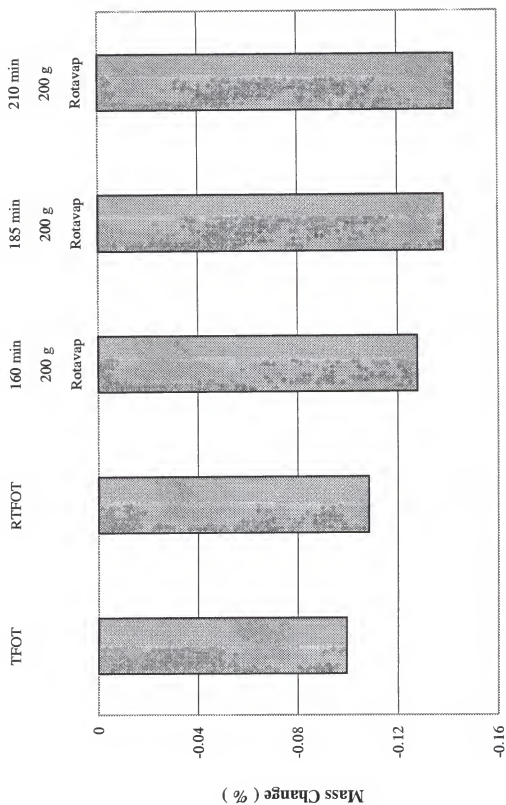
Figure 5.9. Mass change of residues after different aging processes.

As a summary, it seems that using a lower temperature in the Modified Rotavapor aging process to simulate the long-term aging characteristics of asphalt binders will not be practical for two reasons:

1. The asphalt binders will not roll at low temperatures due to their high viscosity

2. The process time required for simulating long-term aging in the Modified Rotavapor aging process may be too long

Therefore, no further evaluation was done to investigate the long-term aging characteristics of asphalt binders in the Modified Rotavapor aging process.

5.3 Final Evaluation of the Improved Rotavapor Aging Apparatus with Morton Flask

5.3.1 Parametric experimental study

From the results of the preliminary study on the aging effects of the Modified Rotavapor Aging apparatus using the Morton flask, the three testing configurations (discussed in 5.2.2) which produced aging severity closest to TFOT and RTFOT were as follows:

1. 163 °C, 200 g and 160 minutes

2. 163 °C, 200 g and 185 minutes

3. 163 °C, 200 g and 210 minutes

In the final evaluation of the improved aging procedure, a total of four asphalt binders (AC-20, AC-30, AC-30+5%CR and AC-30+12%CR) were aged in the Modified Rotavapor Aging apparatus using Morton flask based on these three testing

configurations. It was previously mentioned that no further study will be conducted to simulate long-term field aging in the Modified Rotavapor Aging Process. Thus, the final evaluation was conducted only to simulate short-term aging characteristics of asphalt binders in the Modified Rotavapor Aging Process.

In order to obtain a good estimate of the variability of the aging procedure, ten replicate samples per condition were used for AC-30 and AC-30+5%CR. For AC-20 and AC-30+12%CR, only three samples per condition were used. All four asphalt binders were also subjected to the standard TFOT and RTFOT procedures. Characterization of the aged residues were made using the following tests:

1. Standard Penetration Test at 25 °C

2. Brookfield Viscosity Test at 60 °C

3. Dynamic Shear Rheometer Test at 25 and 64 °C

4. Mass change determination

The test results of AC-20, AC-30, AC-30+5%CR and AC-30+12%CR residues after different aging processes are shown in Appendices A.18 through A.21, respectively. Penetration at 25 °C, Brookfield viscosity at 60 °C, G* at 25 °C, δ at 25 °C, G* at 64 °C, δ at 64 °C and mass change values of AC-20, AC-30, AC-30+5%CR and AC-30+12%CR after different aging processes are shown in Figures 5.10 through 5.16, Figures 5.17 through 5.23, Figures 5.24 through 5.30 and Figures 5.31 through 5.37, respectively.
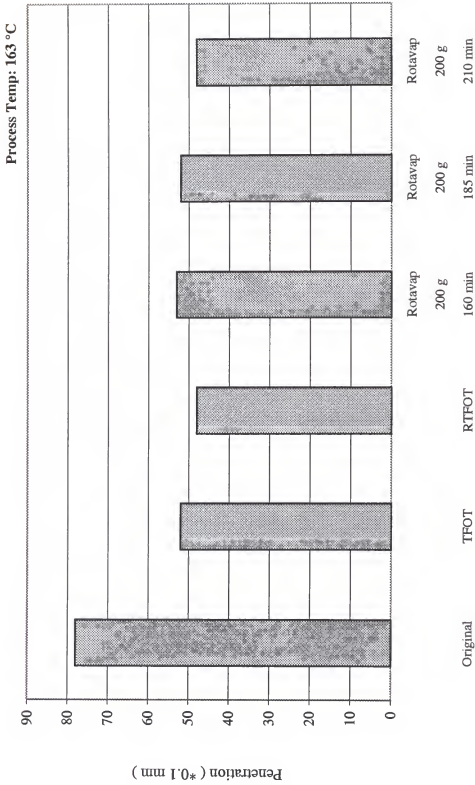
Figure 5.10. Comparison of penetration of AC-20 residues after different aging processes.
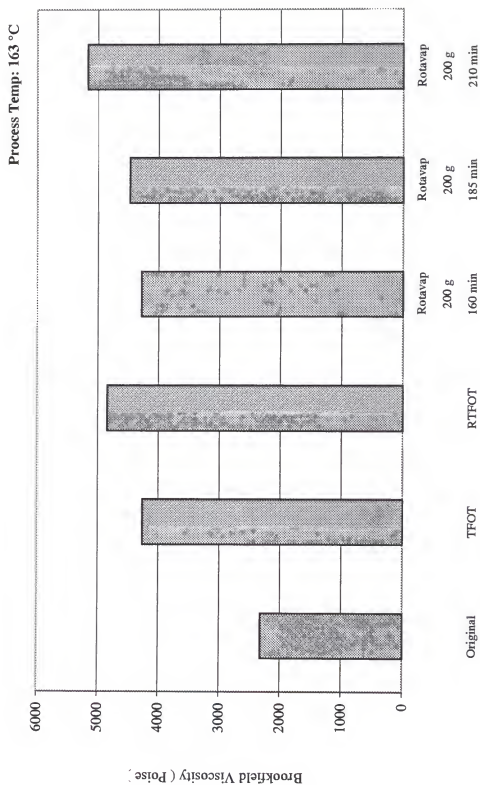
Figure 5.11. Comparison of Brookfield viscosity at 60 °C of AC-20 residues after different aging processes.
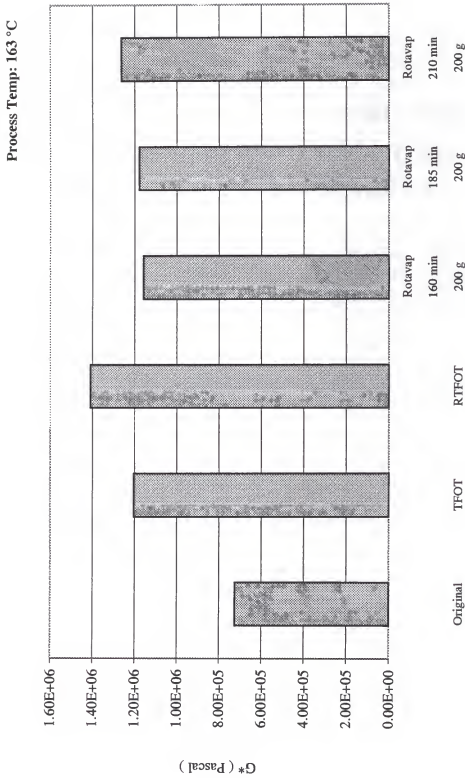
Figure 5.12. Comparison of dynamic modulus (G*) of AC-20 residues at 10 rad/sec and 25 °C after different aging processe.
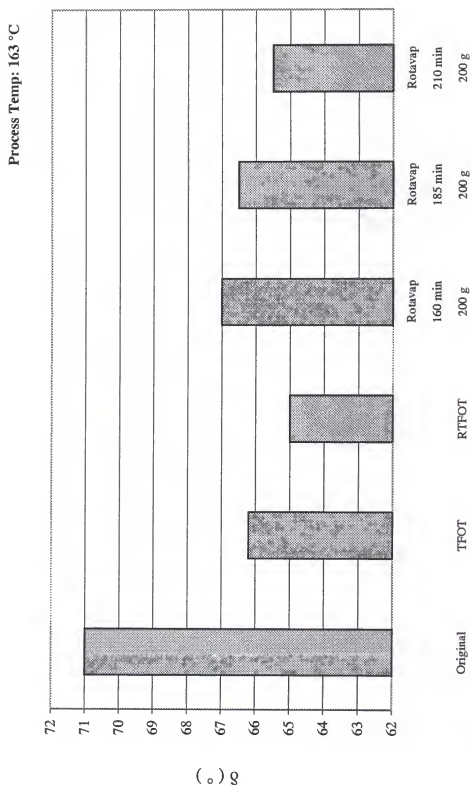
Figure 5.13. Comparison of phase angle (delta) of AC-20 residues at 10 rad/sec and 25 °C after different aging processes.
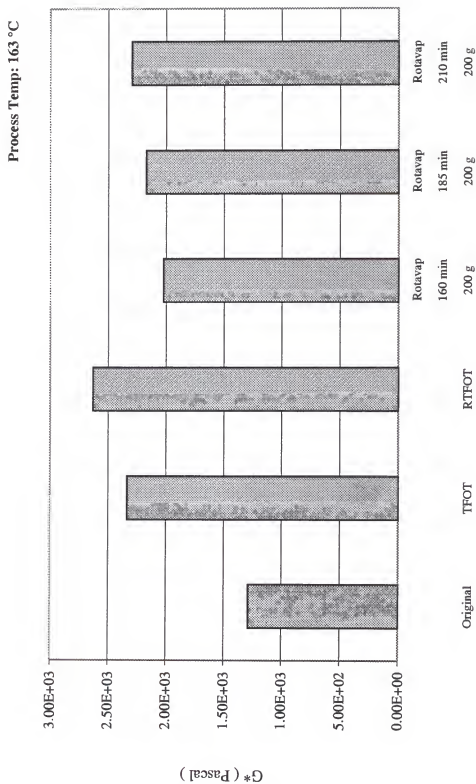
Figure 5.14. Comparison of dynamic modulus (G*) of AC-20 residues at 10 rad/sec and 64 °C after different aging processe.

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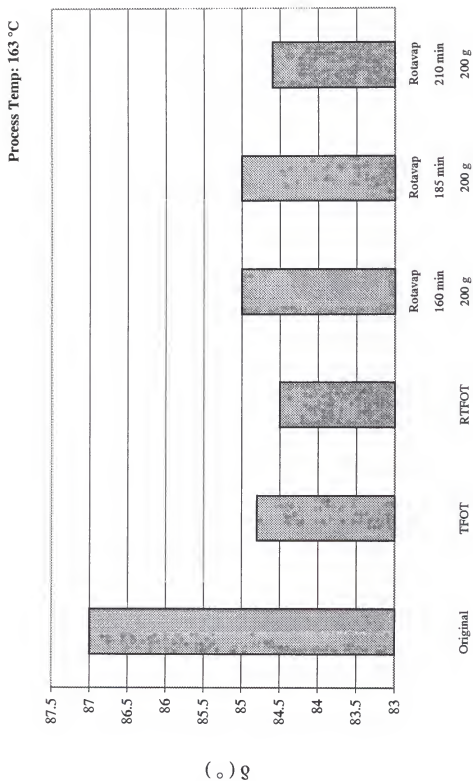
Figure 5.15. Comparison of phase angle (delta) of AC-20 residues at 10 rad/sec and 64 °C after different aging processes.
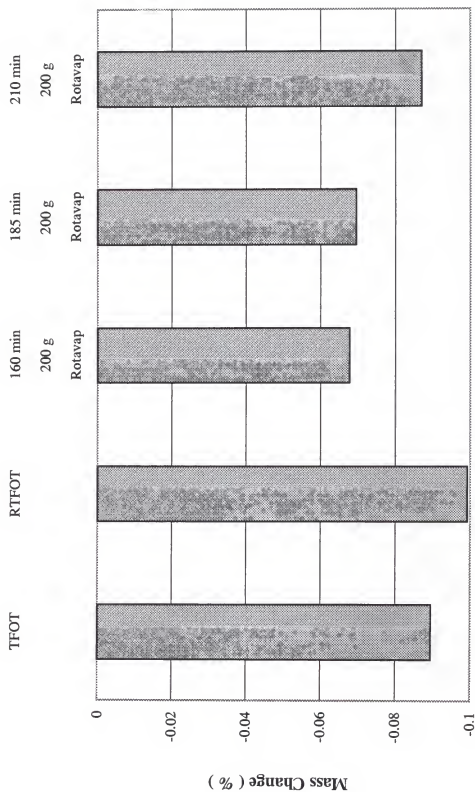
Figure 5.16. Mass change of AC-20 residues after different aging processes.
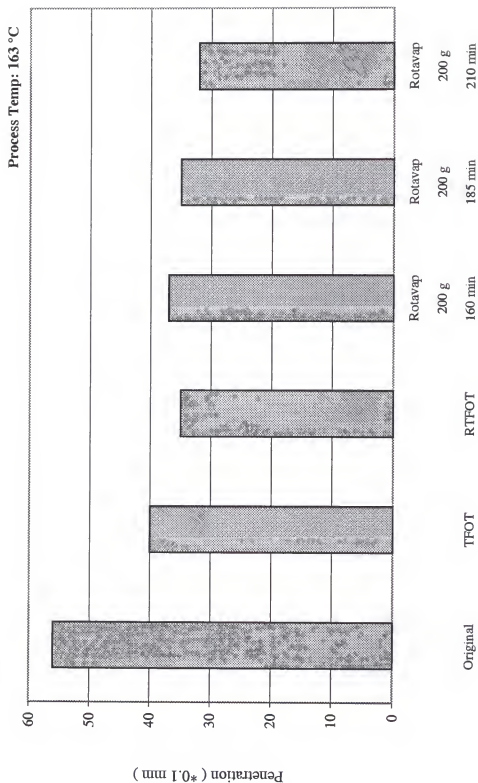
Figure 5.17. Comparison of penetration of AC-30 residues after different aging processes.
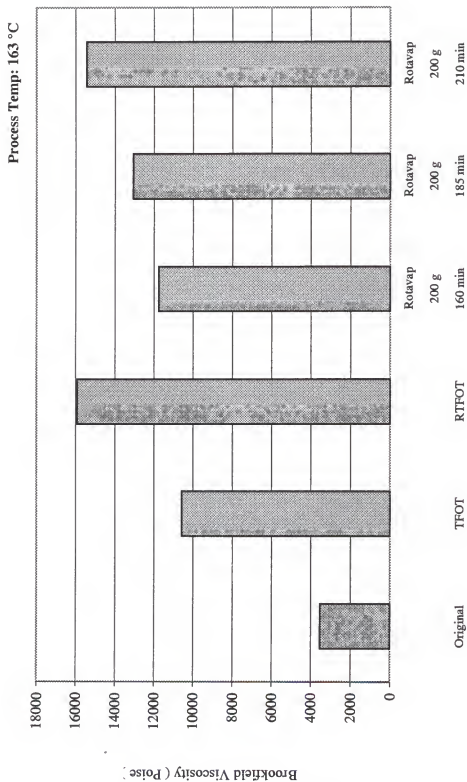
Figure 5.18. Comparison of Brookfield viscosity at 60 °C of AC-30 residues after different aging processes.
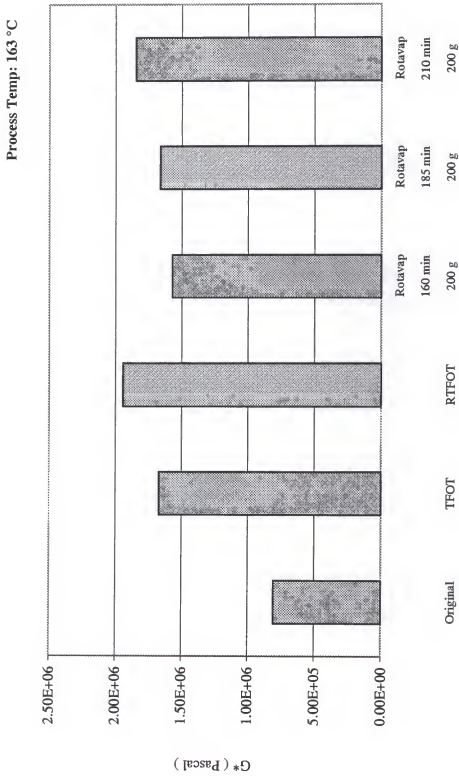
Figure 5.19 Comparison of dynamic modulus (G^*) of AC-30 residues at 10 rad/sec and 25 °C after different aging processes.
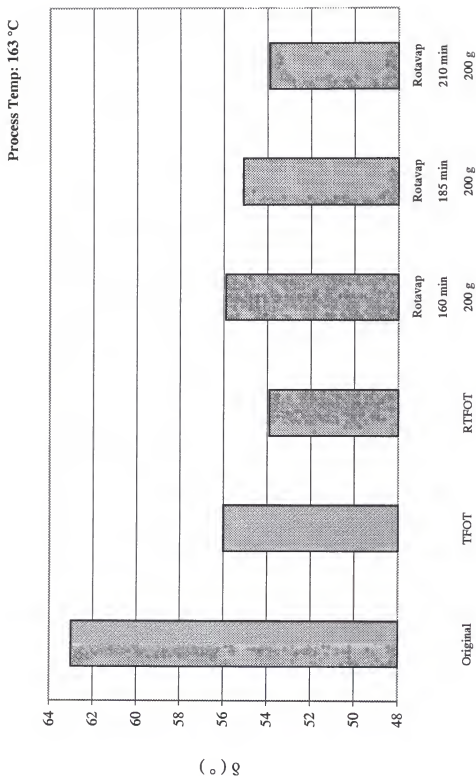
Figure 5.20. Comparison of phase angle (delta) of AC-30 residues at 10 rad/sec and 25 °C after different aging processes.
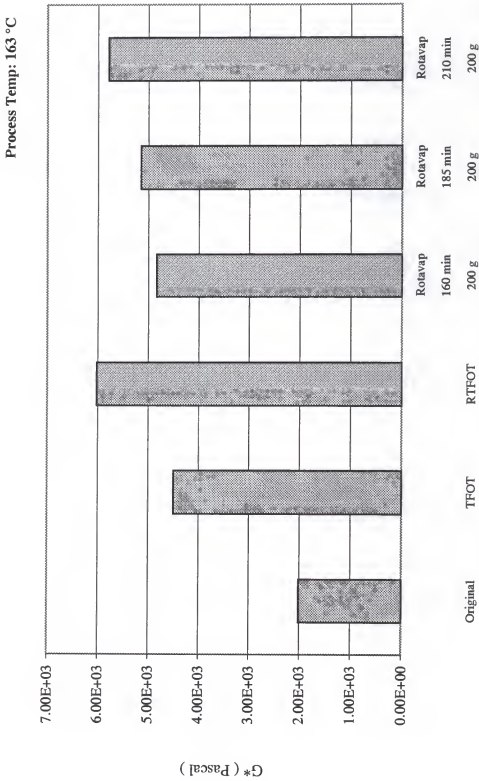
Figure 5.21 Comparison of dynamic modulus ($G*$) of AC-30 residues at 10 rad/sec and 64 °C after different aging processes.
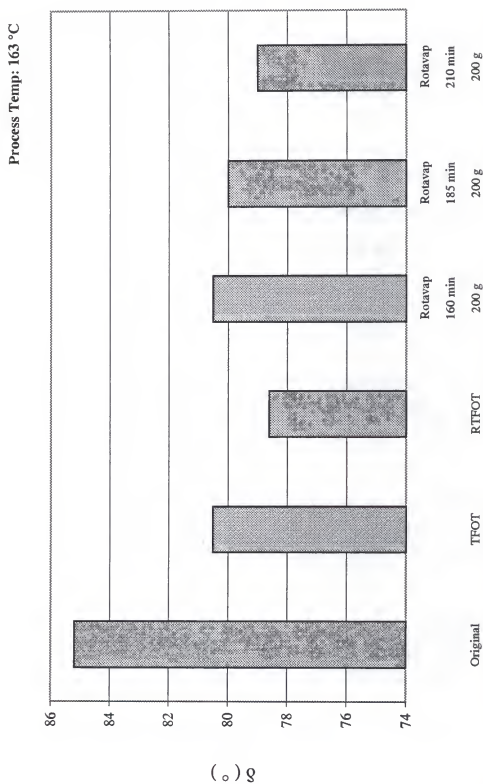
Figure 5.22. Comparison of phase angle (delta) of AC-30 residues at 10 rad/sec and 64 °C after different aging processes.
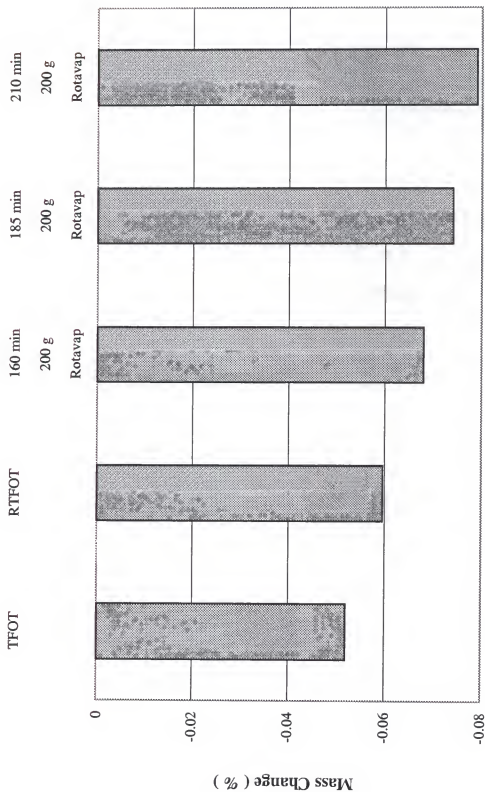
Figure 5.23. Mass change of AC-30 residues after different aging processes.
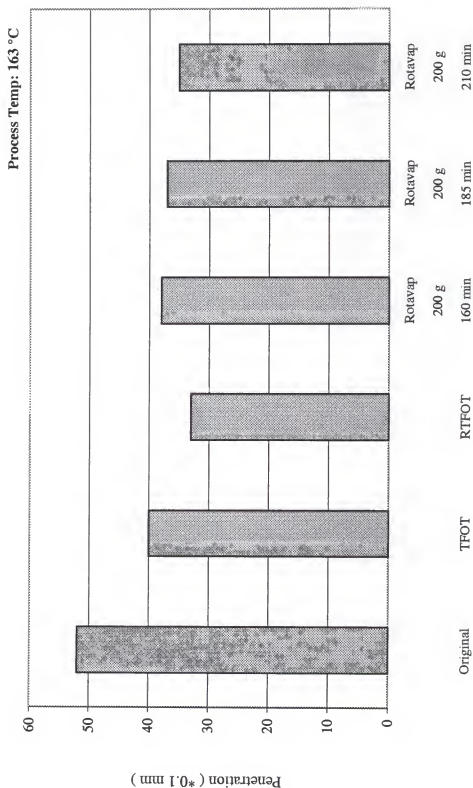
Figure 5.24. Comparison of penetration of AC-30+5%CR residues after different aging processes.
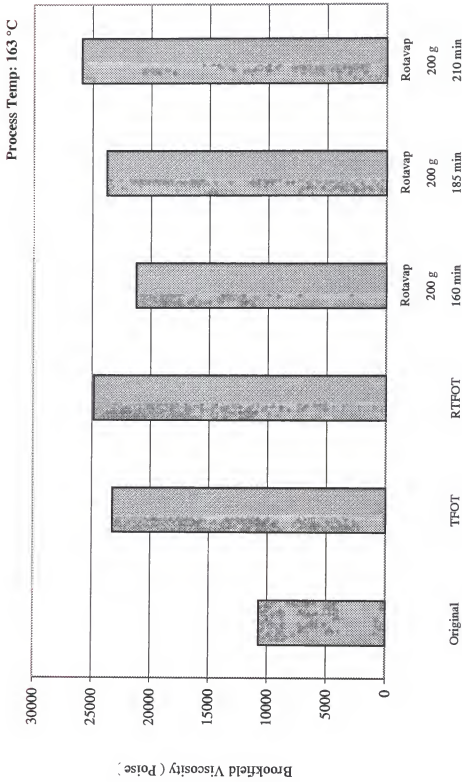
Figure 5.25. Comparison of Brookfield viscosity at 60 °C of AC-30+5%CR residues after different aging processes.
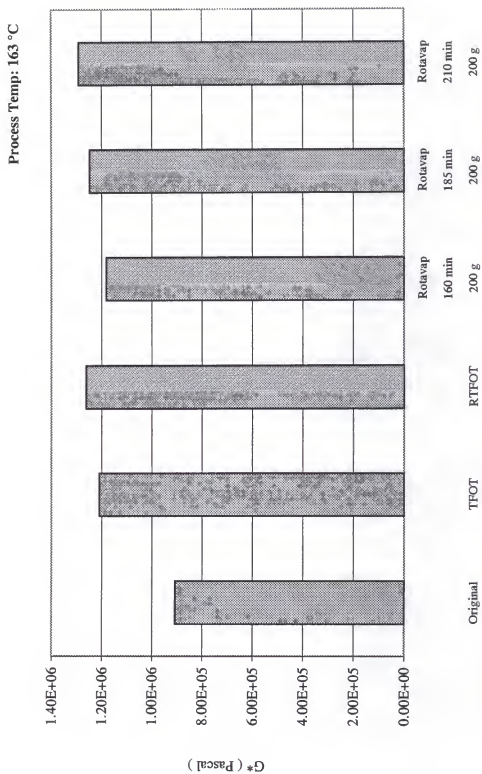
Figure 5.26 Comparison of dynamic modulus (G*) of AC-30+5%CR residues at 10 rad/sec and 25 °C after different aging processes.
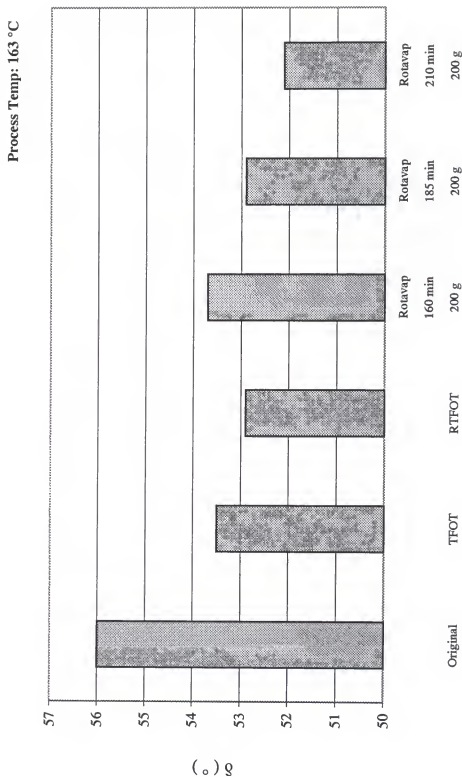
Figure 5.27. Comparison of phase angle (delta) of AC-30+5%CR residues at 10 rad/sec and 25 °C after different aging processes.
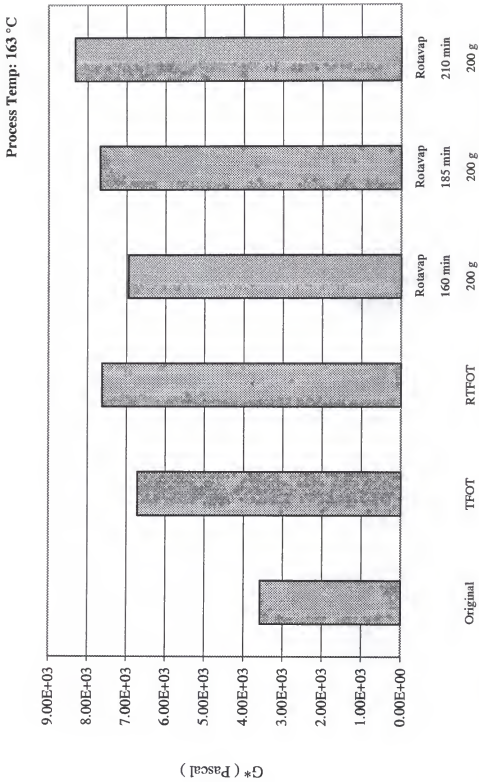
Figure 5.28. Comparison of dynamic modulus (G*) of AC-30+5%CR residues at 10 rad/sec and 64 °C after different aging processes.
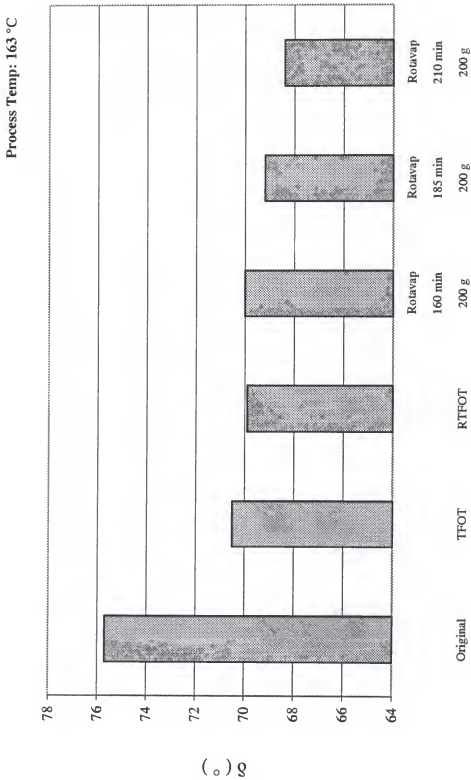
Figure 5.29. Comparison of phase angle (delta) of AC-30+5%CR residues at 10 rad/sec and 64 °C after different aging processes.
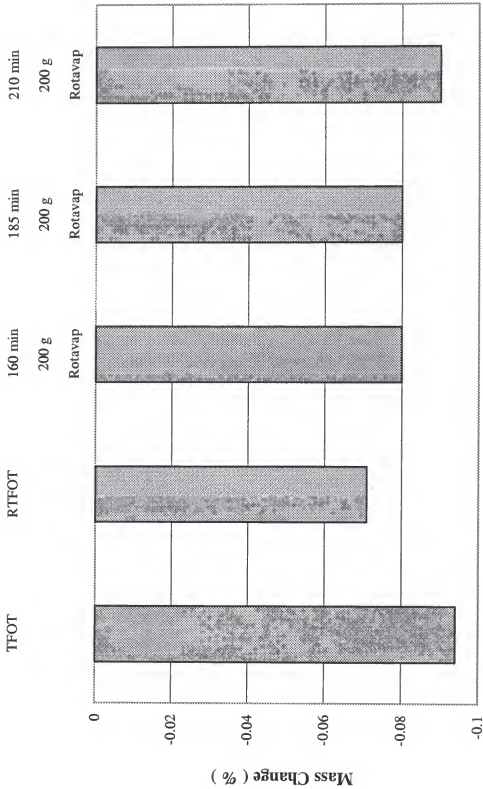
Figure 5.30. Mass change of AC-30+5%CR residues after different aging processes.
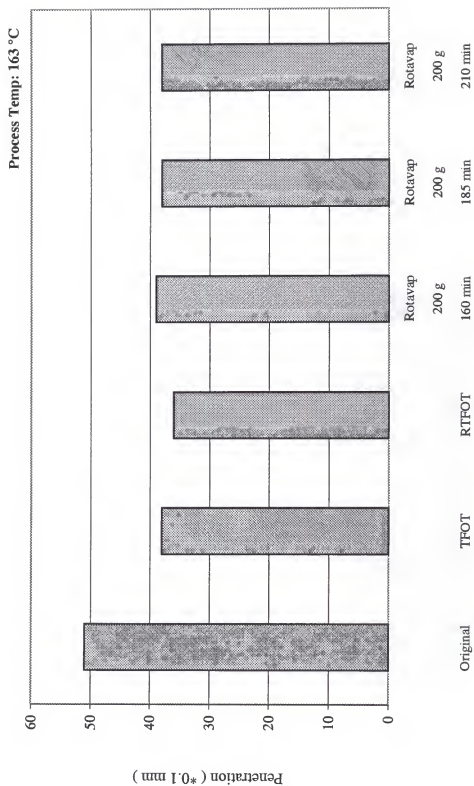
Figure 5.31. Comparison of penetration of AC-30+12%CR residues after different aging processes.
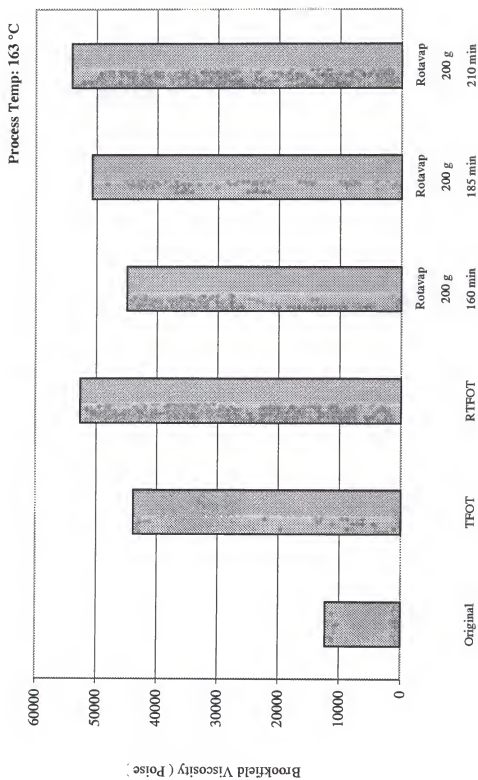
Figure 5.32. Comparison of Brookfield viscosity at 60 °C of AC-30+12%CR residues after different aging processes.
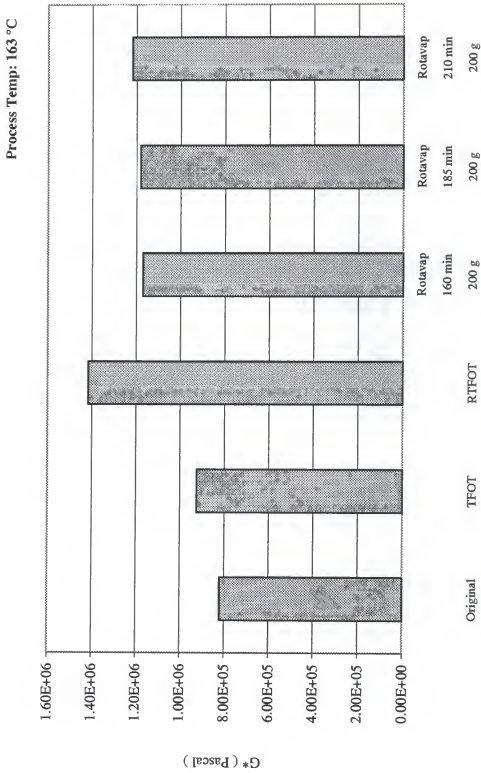
Figure 5.33. Comparison of dynamic modulus (G^*) of AC-30+12%CR residues at 10 rad/sec and 25 °C after different aging processes.
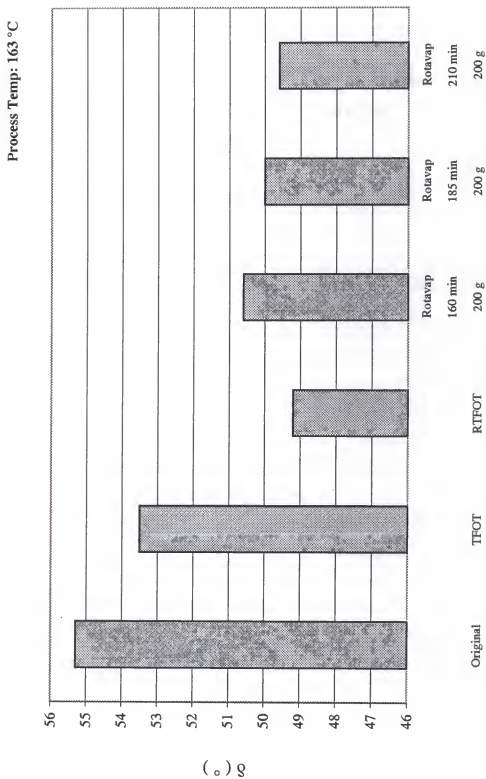
Figure 5.34. Comparison of phase angle (delta) of AC-30+12%CR residues at 10 rad/sec and 25 °C after different aging processes.
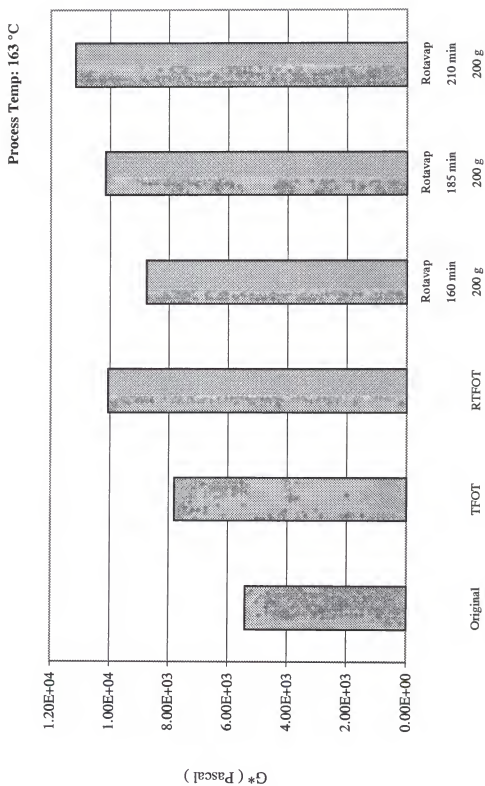
Figure 5.35. Comparison of dynamic modulus (G*) of AC-30+12%CR residues at 10 rad/sec and 64 °C after different aging processes.
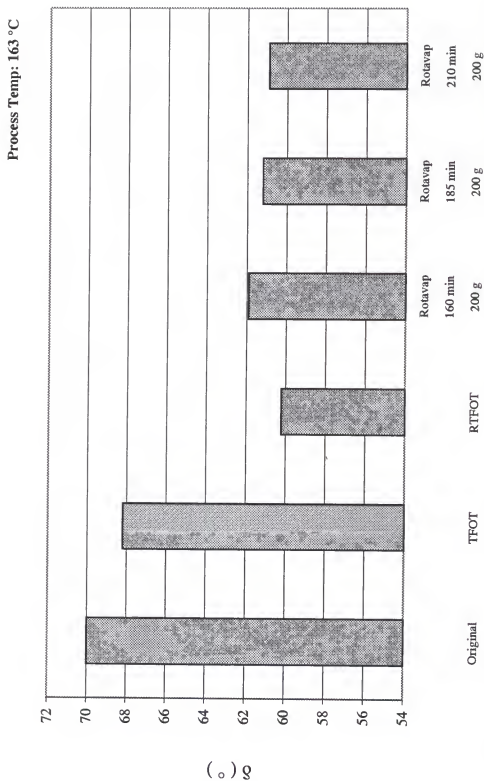
Figure 5.36. Comparison of phase angle (delta) of AC-30+12%CR residues at 10 rad/sec and 64 °C after different aging processes.
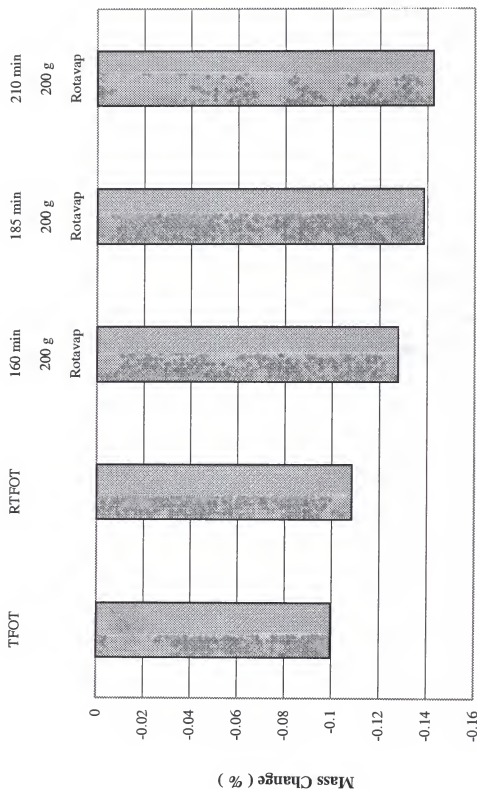
Figure 5.37. Mass change of AC-30+12%CR residues after different aging processes.

5.3.2 Statistical analysis

In order to determine the most appropriate process times to be used in the improved rotavapor aging procedure to produce aging effects similar to the TFOT and the RTFOT, a statistical analysis was conducted on the test results. A linear regression model of the following form was used:

y=a+bx

Where y=test property and x=process time.

Plots of penetration at 25 °C, viscosity at 60 °C, G* at 25 °C, δ at 25 °C, G* at 64 °C, and δ at 64 °C values of the aged AC-20 residues versus process time are shown in Figures 5.38 through 5.43, respectively.

The prediction equations from the regression analysis for the different groups of test properties and asphalt types were used to compute the process times required in the modified rotavapor aging process for producing aging severity closest to TFOT and RTFOT.

The mean values of the test properties of the TFOT and RTFOT aged asphalts were plugged into these prediction equations to solve the required process times for the different groups of data.

In order to exclude unreasonable results, the following criterias were used for selection of the computed process times to be used:

1. The computed time values have to be within or close to the experimental range (160-210 minutes).

2. Only those regression equations which have less than 5 % coefficient of variation were used.
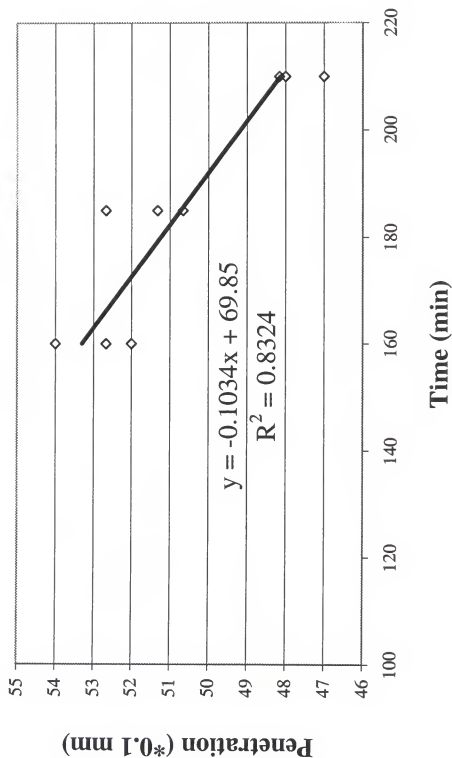
Figure 5.38. Plot of penetration at 25 °C of aged AC-20 residues versus process time.
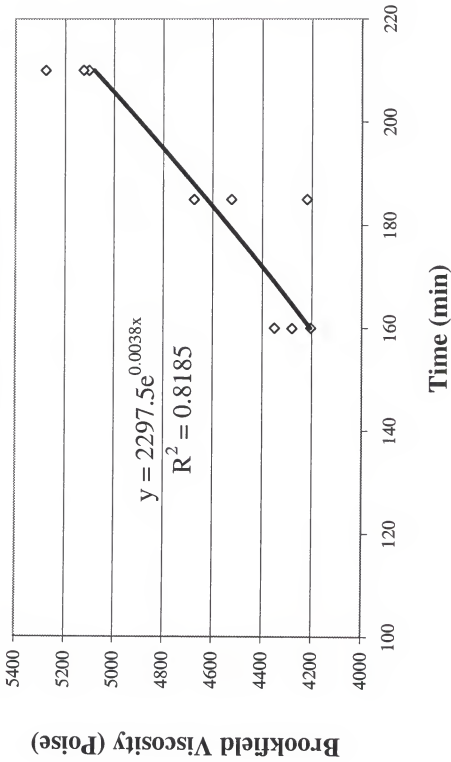
Figure 5.39. Plot of Brookfield viscosity at 60 °C of aged AC-20 residues versus process time.
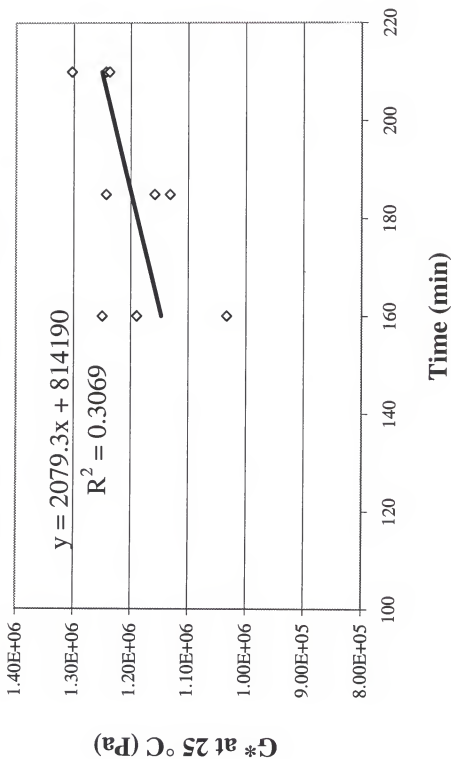
Figure 5.40. Plot of G* at 25 °C of aged AC-20 residues versus process time.
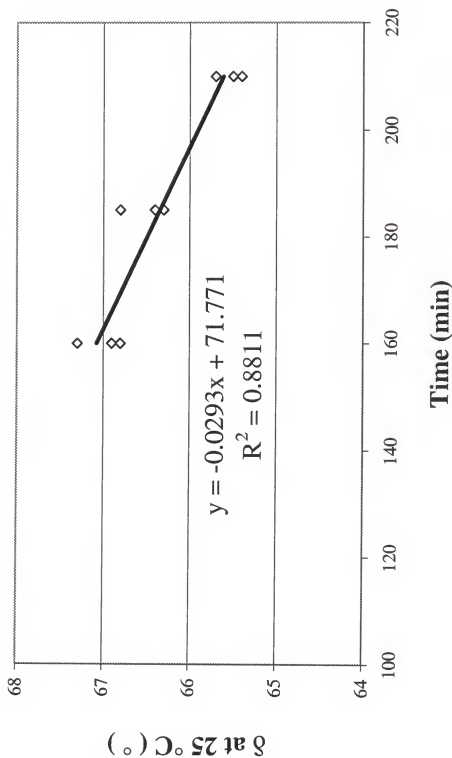
Figure 5.41. Plot of delta at 25 °C of aged AC-20 residues versus process time.
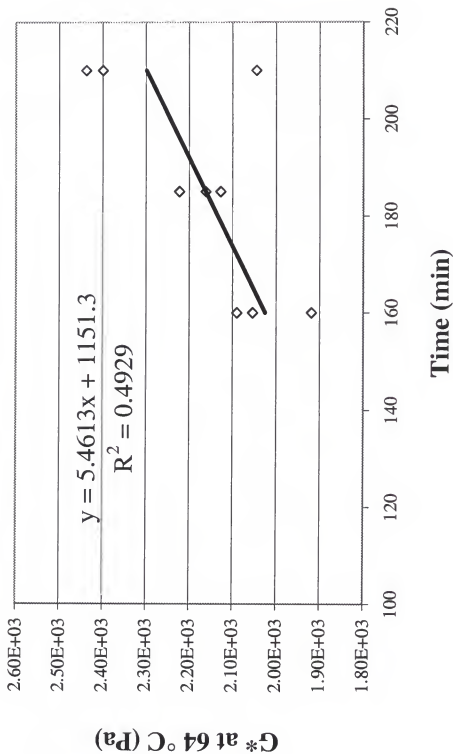
Figure 5.42. Plot of G* at 64 °C of aged AC-20 residues versus process time .
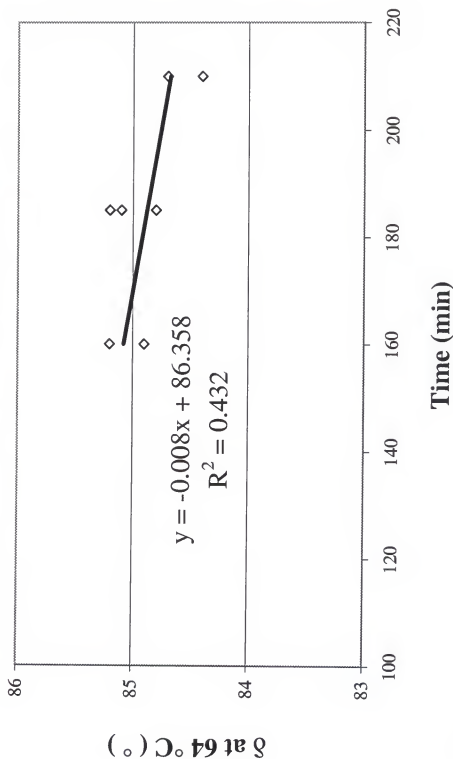
Figure 5.43. Plot of delta at 64 °C of aged AC-20 residues versus process time.

The computed time values obtained in this fashion are displayed in Table 5.1. These time values were then averaged to obtain the average process times for matching with the TFOT and the RTFOT results. It can be seen that an average process time of 165 minutes in the modified rotavapor aging process will produce aging severity closest to TFOT. For producing aging severity closest to RTFOT, an average process time of 210 minutes in the modified rotavapor aging process is needed.

In conclusion, the modified rotavapor aging process at 163 °C, 200 g sample and 165 minutes of process time can produce aging severity similar to TFOT. To produce aging severity similar to RTFOT, the modified rotavapor aging process at 163 °C, a sample weight of 200 g and 210 minutes of process time can be used.

Table 5.1. Computed time values required in the modified rotavapor aging process for producing aging severity closest to TFOT and RTFOT.

Test Type	Asphalt Type	R square	C.V. (%)	Time (TFOT)	Time (RTFOT)
			(Root MSE/Mean) x100	min	min
Pen. At 25 °C	AC-20	0.83	2.11	176	210
	AC-30	0.46	5.67	-	-
	AC-30+5%CR	0.68	2.65	132	235
	AC-30+12%CR	0.012	4.25	-	-
Visc. at 60 °C	AC-20	0.82	4.16	164	196
	AC-30	0.74	7.00	-	-
	AC-30+5%CR	0.21	14.4	-	-
	AC-30+12%CR	0.35	11.3	-	-
G* at 25 °C	AC-20	0.31	6.03	-	-
	AC-30	0.27	11.1	-	-
	AC-30+5%CR	0.14	9.2	-	-
	AC-30+12%CR	0.03	11.5	-	-
δ at 25 °C	AC-20	0.88	0.37	191	232
	AC-30	0.68	1.04	158	212
	AC-30+5%CR	0.73	0.77	165	186
	AC-30+12%CR	0.19	1.97	-	
G* at 64 °C	AC-20	0.49	5.93	-	-
	AC-30	0.44	8.57	-	-
	AC-30+5%CR	0.60	6.32	-	-
	AC-30+12%CR	0.4	13.6	-	-
δ at 64 °C	AC-20	0.43	0.25	199	237
	AC-30	0.66	0.56	163	227
	AC-30+5%CR	0.37	1.25	144	162
	AC-30+12%CR	0.13	2.02	-	-
Average Time				**165 min**	**210 min**

CHAPTER 6
APPLICATION OF THE MODIFIED ROTAVAPOR AGING PROCESS IN THE SUPERPAVE BINDER SPECIFICATIONS

6.1 Introduction

The test results as presented in chapter 5 indicate that the modified rotavapor aging process at 163 °C, for 210 minutes and using a sample weight of 200 g produces aging severity closest to the RTFOT. To evaluate the applicability of the Modified Rotavapor aging process in place of the RTFOT in the Superpave Binder specifications, two asphalt binders (AC-30 and AC-30+12%CR) were graded according to the Superpave Binder specifications by using:

1. RTFOT process

2. Modified Rotavapor aging process

This chapter presents the results of this series of tests.

6.2 Determining the PG Grades of Asphalts

Two asphalt binders (AC-30 and AC-30+12%CR) were subjected to the following short-term aging processes to determine their PG grades according to the Superpave Binder specifications.

1. RTFOT

2. Modified Rotavapor aging process in place of RTFOT

After the binders were aged in these two short-term aging processes, they were subjected to the PAV process at 100 °C to simulate the long-term field aging. Characterization of the asphalt binders were made using the following tests:

1. Brookfield Viscosity Test at 135 °C on original asphalt binders.

2. Mass Change Determination using RTFOT and Modified Rotavapor(ModRvap).

3. Dynamic Shear Rheometer (DSR) test at high temperatures on original and short-term aged asphalt residues.

4. Dynamic Shear Rheometer (DSR) test at intermediate temperatures on PAV aged asphalt residues.

5. Bending Beam Rheometer (BBR) test on PAV aged asphalt residues.

6.2.1 Test results and discussion

6.2.1.1 Brookfield viscosity test at 135 °C

In order to provide good pumping and handling of asphalt binders in mixing plants, it is required in the specification that the original asphalt binder must have a maximum viscosity of 3 Pa.s at 135 °C. It can be seen from Table 6.1 that both AC-30 and AC-30+12%CR have satisfied the criteria by having a viscosity at 135 °C of less than 3 Pa.s.

Table 6.1. Brookfield viscosity and mass change results of AC-30 and AC-30+12%CR.

Asphalt Type	Replicate	Viscosity@ 135 °C	Mass Change (%)	
		(Pa.s)	RTFOT	ModRvap
AC-30	1	0.914	-0.0598	-0.082
	2	0.8	-0.0605	-0.075
	Average	**0.857**	**-0.06**	**-0.078**
SR-580(AC-30+12%CR)	1	2.2	-0.1089	-0.144
	2	2.2	-0.1072	-0.141
	Average	**2.2**	**-0.108**	**-0.142**

6.2.1.2 Mass change determination

Excessive aging (volatilization) during the hot-mixing and construction process is controlled by limiting the maximum mass loss to 1 % for the short-term aged asphalt residues in the Superpave Binder specifications. In this study, both asphalts were subjected to the RTFOT as well as the Modified Rotavapor aging process to determine the amount of mass changes. Table 6.1 shows that both AC-30 and AC-30+12%CR have experienced mass loss less than 1 % in the two short-term aging processes. It can also be seen that the Modified Rotavapor aging process produces a mass loss very close to those of RTFOT.

6.2.1.3 DSR test at high temperatures

The Superpave Binder specification controls the rutting resistance at high pavement service temperatures by measuring the stiffness of asphalt binders. The rutting factor, $G^*/\sin \delta$, must be 1000 Pa or more for original asphalt binders and at least 2200 Pa for the short-term aged asphalt binders at the maximum pavement design temperature to control rutting.

To determine the high temperature part of their PG grades, both AC-30 and AC-30+12%CR were subjected to the RTFOT and the Modified Rotavapor aging processes. The DSR test results of original and short-term aged asphalt binders at high temperatures are given in Table 6.2. The conventional asphalt binder (AC-30, AC-30 aged in the RTFOT and AC-30 aged in the ModRvap) samples were tested at 58, 64 and 70 °C in the DSR. Three samples per test temperature were tested. The test results indicated that both AC-30 aged in the RTFOT and in the ModRvap aging process have failed at 70 °C. Therefore, their starting PG temperature is 64 °C.

The standard deviations of G*/Sin δ values of the aged residues after the RTFOT and those after the modified rotavapor process are also shown in Table 6.2. It can be seen that the variation after the RTFOT process is higher than that after the modified rotavapor aging process.

For AC-30+12%CR, DSR tests at 58, 64, 70, 76 and 82 °C were run on AC-30+12%CR, AC-30+12%CR aged in the RTFOT and AC-30+12%CR aged in the ModRvap aging process. It can be seen from Table 6.2 that AC-30+12%CR both aged in the RTFOT and in the ModRvap aging process have satisfied the rutting criteria at all temperatures. Thus, the starting PG grade temperature for AC-30+12%CR (both aged in the RTFOT and in the ModRvap aging process) is 82 °C.

The comparison of standard deviations for AC-30+12%CR show that the variation is sometimes higher in the RTFOT and sometimes higher in the ModRvap process depending on the testing temperature.

The comparison between the two procedures (RTFOT and the modified rotavapor aging process) was obtained by plotting G*/Sin δ values for AC-30 and AC-30+12%CR

aged residues. Figure 6.1 shows that there is a good correlation between the G*/Sin

δ after the RTFOT and G*/Sin δ after the ModRvap when one data point was excluded.

6.2.1.4 DSR test at intermediate temperatures

The fatigue cracking in asphalt pavements is controlled by using a maximum limit

of 5000 kPa for G* Sin δ value of PAV aged residues in the DSR test at intermediate

temperatures.

After determining the starting PG temperatures of AC-30 and AC-30+12%CR,

RTFOT+PAV and ModRvap+PAV aged asphalt residues were tested in the DSR to

determine the temperatures at which the fatigue cracking criteria is satisfied.

It can be seen from Table 6.3 that AC-30(RTFOT+PAV) aged residues have

failed at 19 °C and AC-30(ModRvap+PAV) aged residues have failed at 16 °C. The

variation is sometimes higher in the RTFOT and sometimes higher in the ModRvap

process depending on the testing temperature for AC-30 aged residues.

For the rubber asphalt (AC-30+12%CR), both RTFOT+PAV and ModRvap+PAV

aged residues have satisfied the fatigue cracking criteria at all testing temperatures (37,

34, 31 and 28 °C). The comparison of standard deviations show that the variation was

less in the RTFOT process than that of the ModRvap process for rubber asphalt residues.

Figures 6.2 and 6.3 show that there is a good correlation between G* Sin δ after

RTFOT+PAV and G* Sin δ after the ModRvap+PAV for AC-30 and AC-30+12%CR

aged residues, respectively.

Table 6.2. DSR test results of asphalts at high temperatures.

Asphalt Type	Temp(°C)	G* / SIN δ (PA)				
		58	64	70	76	82
	Rep 1	5.01E+03	1.97E+03	9.78E+02	-	-
AC-30	Rep 2	4.73E+03	1.90E+03	9.81E+02	-	-
	Rep 3	4.88E+03	1.93E+03	9.97E+02	-	-
	Avg	**4.87E+03**	**1.93E+03**	**9.85E+02**	-	-
	% Diff	2.89	1.89	0.71	-	-
	Rep 1	1.66E+04	6.90E+03	3.40E+03	-	-
AC-30(RTFOT)	Rep 2	1.58E+04	6.27E+03	3.21E+03	-	-
	Rep 3	1.61E+04	6.58E+03	3.14E+03	-	-
	Avg	**1.62E+04**	**6.58E+03**	**3.25E+03**	-	-
	% Diff	2.33	4.84	4.63	-	-
	Std.Dev	400	316	135		
	Rep 1	1.49E+04	6.33E+03	2.86E+03	-	-
AC-30(ModRvap)	Rep 2	1.49E+04	5.72E+03	2.89E+03	-	-
	Rep 3	1.47E+04	5.91E+03	2.83E+03	-	-
	Avg	**1.48E+04**	**5.99E+03**	**2.86E+03**	-	-
	% Diff	0.46	5.76	1.00	-	-
	Std.Dev	89	313	30		
	Rep 1	1.25E+04	5.87E+03	3.43E+03	1.94E+03	1.67E+03
SR-580	Rep 2	1.27E+04	6.06E+03	3.63E+03	1.85E+03	1.65E+03
(AC-30+12%CR)	Rep 3	1.22E+04	6.05E+03	3.28E+03	1.99E+03	1.61E+03
	Avg	**1.25E+04**	**6.00E+03**	**3.45E+03**	**1.93E+03**	**1.65E+03**
	% Diff	1.76	2.05	5.35	4.02	2.10
	Rep 1	2.97E+04	1.45E+04	8.19E+03	4.52E+03	3.93E+03
SR-580(RTFOT)	Rep 2	2.89E+04	1.45E+04	8.32E+03	4.81E+03	3.92E+03
	Rep 3	2.97E+04	1.47E+04	8.28E+03	4.55E+03	3.99E+03
	Avg	**2.94E+04**	**1.46E+04**	**8.26E+03**	**4.63E+03**	**3.94E+03**
	% Diff	1.87	0.36	0.91	3.96	0.60
	Std.Dev	477	95	69	159	37
	Rep 1	3.02E+04	1.48E+04	8.45E+03	4.59E+03	3.93E+03
SR-580(ModRvap)	Rep 2	2.97E+04	1.49E+04	8.25E+03	4.62E+03	3.82E+03
	Rep 3	2.96E+04	1.46E+04	8.66E+03	4.81E+03	3.90E+03
	Avg	**2.99E+04**	**1.48E+04**	**8.46E+03**	**4.67E+03**	**3.88E+03**
	% Diff	0.46	0.78	2.38	2.88	1.52
	Std.Dev	320	144	202	117	53

Table 6.3. DSR test results of asphalts at intermediate temperatures.

G* SIN δ (PA)						
Temperature (°C)		28	25	22	19	16
Asphalt Type						
	Rep 1	2.19E+06	3.07E+06	4.60E+06	6.26E+06	-
AC-30	Rep 2	2.10E+06	3.51E+06	3.73E+06	6.25E+06	-
(RTFOT+PAV)	Rep 3	2.17E+06	3.00E+06	4.06E+06	5.09E+06	-
	Avg	**2.15E+06**	**3.20E+06**	**4.13E+06**	**5.87E+06**	-
	% Diff	2.52	9.90	11.28	13.30	-
	Std.Dev	48003	276295	435555	675389	-
	Rep 1	1.99E+06	3.10E+06	4.07E+06	4.66E+06	6.94E+06
AC-30	Rep 2	1.76E+06	2.83E+06	3.59E+06	4.38E+06	7.47E+06
(ModRvap+PAV)	Rep 3	2.08E+06	2.68E+06	3.53E+06	4.75E+06	6.05E+06
	Avg	**1.94E+06**	**2.87E+06**	**3.73E+06**	**4.60E+06**	**6.82E+06**
	% Diff	9.57	7.94	9.15	4.72	11.29
	Std.Dev	167506	212638	297298	193317	718558
G* SIN δ (PA)						
Temperature (°C)		37	34	31	28	
Asphalt Type						
	Rep 1	4.62E+05	5.41E+05	6.15E+05	1.04E+06	
SR-580	Rep 2	4.19E+05	5.38E+05	6.97E+05	9.29E+05	
(RTFOT+PAV)	Rep 3	4.85E+05	5.63E+05	6.00E+05	9.44E+05	
	Avg	**4.55E+05**	**5.47E+05**	**6.37E+05**	**9.71E+05**	
	% Diff	7.96	2.91	9.40	4.38	
	Std.Dev	33349	13910	52413	60625	
	Rep 1	4.32E+05	4.97E+05	6.96E+05	9.33E+05	
SR-580	Rep 2	4.29E+05	5.63E+05	6.18E+05	7.43E+05	
(ModRvap+PAV)	Rep 3	3.69E+05	5.55E+05	5.85E+05	8.23E+05	
	Avg	**4.10E+05**	**5.38E+05**	**6.33E+05**	**8.33E+05**	
	% Diff	10.05	7.74	9.97	11.98	
	Std.Dev	35700	36323	56985	95212	
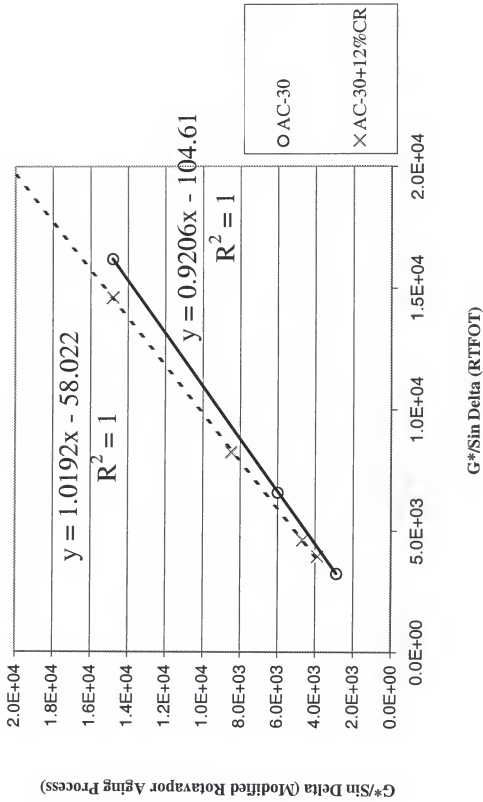
Figure 6.1. Plot of G*/sin delta (RTFOT) versus G*/sin delta (ModRvap).
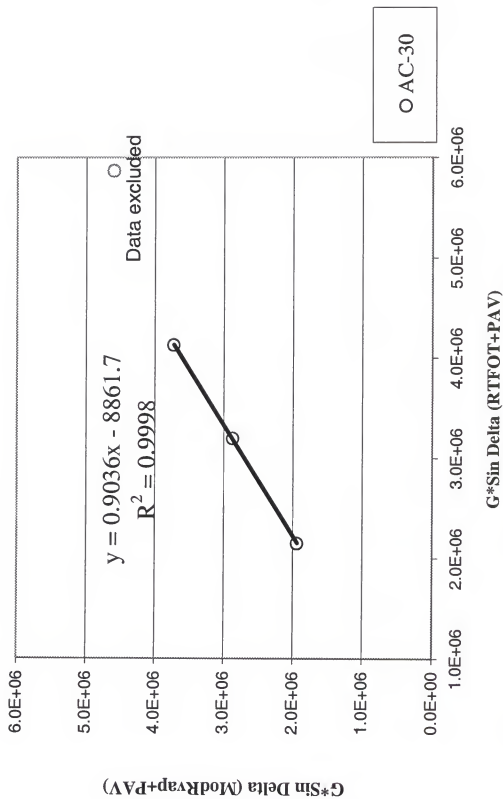
Figure 6.2. Plot of G*/sin delta (RTFOT+PAV) versus G*/sin delta (ModRvap+PAV) for AC-30.
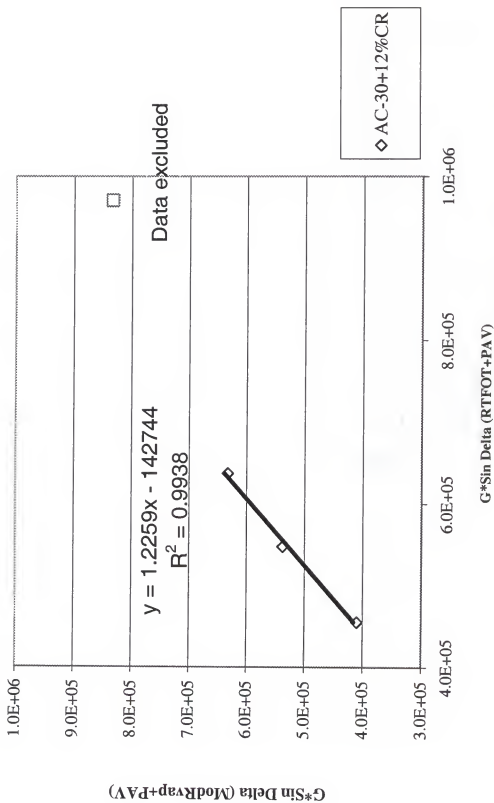
Figure 6.3. Plot of G*sin delta (RTFOT+PAV) versus G*sin delta (ModRvap+PAV) for AC-30+12%CR.

6.2.1.5 Bending Beam Rheometer Test

To determine the lowest pavement design temperature, asphalt binders are tested in the Bending Beam Rheometer(BBR) according to the Superpave Binder specifications. This test is used to control low temperature cracking by limiting the maximum creep stiffness(S) to 300 MPa and minimum m-value(slope) to 0.3.

The results of BBR tests on long-term aged asphalt residues are given in Table 6.4. It can be seen from Table 6.4 that the low temperature cracking criteria for AC-30 after RTFOT+PAV, AC-30 after ModRvap+PAV, AC-30+12%CR after RTFOT+PAV and AC30+12%CR after ModRvap+PAV are satisfied at the lowest temperature of −12 °C. All samples have failed at −24 and −18 °C.

The comparison of standard deviations show that the variation is sometimes higher in the RTFOT and sometimes higher in the ModRvap process depending on the testing temperature based on S and m-values in the BBR test.

Table 6.4. Bending beam rheometer test results of asphalts.

Temperature (°C)		-24		-18		-12	
		S	Slope	S	Slope	S	Slope
Asphalt Type		(Mpa)	m-value	(Mpa)	m-value	(Mpa)	m-value
AC-30(RTFOT+PAV)	Rep 1	-	-	276	0.273	134	0.321
	Rep 2	-	-	272	0.274	132	0.325
	Avg	**-**	**-**	**274**	**0.274**	**133**	**0.323**
	% Diff	-	-	0.73	0.18	0.75	0.62
	Std.Dev			2.83	0	1.41	0
AC-30(ModRvap+PAV)	Rep 1	-	-	256	0.282	121	0.330
	Rep 2	-	-	252	0.282	118	0.334
	Avg	**-**	**-**	**254**	**0.282**	**120**	**0.332**
	% Diff	-	-	0.79	0.000	1.26	0.60
	Std.Dev			2.83	0	2.12	0
SR-580(RTFOT+PAV)	Rep 1	330	0.238	167	0.285	75	0.334
	Rep 2	327	0.241	153	0.296	76	0.330
	Avg	**329**	**0.240**	**160**	**0.291**	**76**	**0.332**
	% Diff	0.46	0.63	4.38	1.89	0.53	0.60
	Std.Dev	2.12	0	9.90	0	0.57	0
SR-580(ModRvap+PAV)	Rep 1	319	0.253	153	0.301	70	0.345
	Rep 2	314	0.256	153	0.294	76	0.332
	Avg	**317**	**0.255**	**153**	**0.298**	**73**	**0.339**
	% Diff	0.79	0.59	0.00	1.18	4.11	1.92
	Std.Dev	3.54	0	0	0	4.24	0

6.2.2 Selection of the PG grade

The binder test results for AC-30(aged in the RTFOT and in the ModRvap) and AC-30+12%CR(aged in RTFOT and in ModRvap) are summarized in Table 6.5. It can be seen from the table that all Superpave Binder Specification requirements for both AC-30(RTFOT) and AC-30(ModRvap) are satisfied at the maximum pavement design temperature of 64 and the minimum pavement design temperature of –22. Therefore, both are graded as PG64-22. For AC-30+12%CR(aged in the RTFOT) and AC-30+12%CR (aged in the ModRvap), all requirements are satisfied at a maximum temperature of 82 and a minimum temperature of –22 °C. Thus, they are graded as PG82-22.

In conclusion, using the Modified Rotavapor aging process in place of the RTFOT in the Superpave Binder Specifications did not affect the PG grades of both the conventional and the rubber modified asphalts used in this testing program.

Table 6.5. PG grades of asphalts after different aging processes.

Asphalt Type	PG Grade	Mass Change(%)	Brookfield Visc. Viscosity	DSR @ High Temp G*/ Sin δ	DSR @ int. Temp G* Sin δ	BBR S	BBR m	Comment
AC-30	PG70-28	P	P	F	P	P	F	F
(Aged in RTFOT)	PG64-28	P	P	P	P	P	F	F
	PG64-22	**P**	**P**	**P**	**P**	**P**	**P**	**P**
AC-30	PG70-28	P	P	F	P	P	F	F
(Aged in ModRvap)	PG64-28	P	P	P	P	P	F	F
	PG64-22	**P**	**P**	**P**	**P**	**P**	**P**	**P**
SR-580(AC-30+12%CR)	PG82-34	P	P	P	P	F	F	F
(Aged in RTFOT)	PG82-28	P	P	P	P	P	F	F
	PG82-22	**P**	**P**	**P**	**P**	**P**	**P**	**P**
SR-580(AC-30+12%CR)	PG82-34	P	P	P	P	F	F	F
(Aged in ModRvap)	PG82-28	P	P	P	P	P	F	F
	PG82-22	**P**	**P**	**P**	**P**	**P**	**P**	**P**

P-Pass, F-Fail

CHAPTER 7
SUMMARY OF FINDINGS AND RECOMMENDATIONS

7.1 Summary of Findings

A research study was conducted to develop a suitable laboratory aging procedure to simulate hot-mix plant aging on modified asphalts. The application of the developed method in the Superpave Binder Specifications was also investigated. The summary of the main findings from this study are as follows:

1. The Rotavapor apparatus was successfully modified to work as an aging device for both conventional and modified asphalts. The test results indicated that the aging severity of the modified rotavapor aging process is affected by variables such as process temperature, process duration and sample size. Smaller sample size, longer process duration and higher process temperature produce significantly more severe aging effects.

2. The temperature variation in the oil bath of the rotavapor apparatus was substantially reduced by covering the oil bath with a well-insulated box.

3. The Modified Rotavapor Aging process at 163 °C, 200 g sample size and 160 minutes duration produces aging severity similar to that of the TFOT, while the Modified Rotavapor aging process at the test configuration of 163 °C, 200 g and 210 minutes produces aging severity similar to the RTFOT.

4. Based on limited data, to produce aging severity similar to TFOT+PAV or RTFOT+PAV, the Modified Rotavapor aging process can be run at 163 °C for 615 minutes using a sample size of 125 g.

5. The rubber asphalt in the rubber asphalt mixture could not be completely extracted in the reflux extraction process. Most of the rubber particles remain on the filter paper and stick to the aggregates during the extraction process. A small amount of asphalt binder also remains in the aggregate and can not be completely extracted. The percent of rubber and asphalt remaining on the filter paper in the reflux extraction process is different for each mixture.

6. The extracted aggregate from the Reflux extraction should be left in a dessicator to cool to room temperature after drying in an oven. The weight of the extracted aggregate stabilizes after 3 hours in a dessicator and thus represents its true weight at room temperature. Therefore, this weight should be used for calculating the extracted AC content to eliminate the possible effects of moisture in air on the results.

7. All asphalt binders aged in the Modified Rotavapor aging process have experienced a mass loss very close to those aged in the TFOT and RTFOT processes.

8. The PG grades of both the conventional and the rubber modified asphalts used in this study were not affected by using the Modified Rotavapor Aging process in place of the RTFOT in the Superpave Binder Specifications.

9. The results from this study also verify the prior finding that the RTFOT is a more severe short-term aging process than that of TFOT.

7.2 Recommendations

Some recommendations for a future study are as follows:

1. Different modified asphalts such as SBR, SBS modified asphalts could be tested in the Modified Rotavapor aging process to validate the results obtained from this study.

2. Further investigation on the extraction of rubber asphalt binders from rubber asphalt mixtures is needed to compare the aging of the binder in the modified rotavapor process with that in the asphalt mixtures in service.

APPENDIX

Table A1. Penetration at 25 °C.

Penetration (*0.1 mm)					
Rotavapor aging process					
Binder		AC-30		AC-30+10%CR	
Process Time	Process Temp	Sample Size		Sample Size	
		50 g	75 g	50 g	75 g
60 min	163 °C	-	-	41.3	42.5
	185 °C	-	-	36.3	41.2
85 min	163 °C	42.8	-	40	42.3
	185 °C	35.2	-	38	37.5
Standard short-term aging process					
Binder		AC-30		AC-30+10%CR	
Test	Process Temp	Penetration (*0.1 mm)			
TFOT	163 °C	45.7		37.3	
RTFOT	163 °C	39.8		37.2	

Table A2. Brookfield viscosity at 60 °C.

Brookfield Viscosity (Poise)					
Rotavapor aging process					
Binder		AC-30		AC-30+10%CR	
Process Time	Process Temp	Sample Size		Sample Size	
		50 g	75 g	50 g	75 g
60 min	163 °C	-	-	19998	16483
	185 °C	-	-	23565	23374
85 min	163 °C	7485	-	20183	18484
	185 °C	12738	-	25814	27811
Standard short-term aging process					
Binder		AC-30		AC-30+10%CR	
Test	Process Temp	Brookfield Viscosity (Poise)			
TFOT	163 °C	7111		18365	
RTFOT	163 °C	9508		24096	

Table A3. Temperature measurements (°C).

Vertical Distance from Oil Surface (cm)	Temp. with Insulated Box (°C)	Temp. without Insulated Box (°C)
-8	181	177
-6	181	176
-4	181	171
-2	180	142
0	156	95
2	148	65
Horizontal Distance from Center (cm)	Temp. with Insulated Box (°C)	Temp. without Insulated Box (°C)
-13	181	177
-6.5	181	177
0	181	177
6.5	181	177
13	181	177

Table A4. Penetration at 25 °C.

Modified Rotavapor aging process						
Penetration (*0.1 mm)						
Binder		AC-30			AC-30+10%GTR		
Sample Size		50 g	125 g	200 g	50 g	125 g	200 g
Process Time	Replicate						
60 min	R1	41.7	48.3	53.7	43.3	45	46.7
	R2	43.6	50	54.7	44.3	44	45.3
85 min	R1	41.5	47.5	51.3	40.3	41.7	43.3
	R2	42	47	51	42.2	41.7	43.2
110 min	R1	35.3	45.3	48.3	35.3	35	37.7
	R2	35.7	45.3	49.3	35.3	37.3	37.3
Standard short-term aging processes						
Penetration (*0.1 mm)						
Binder		AC-30			AC-30+10%CR		
Test	Replicate						
TFOT	R1	45.3			41.7		
	R2	45.7			41		
RTFOT	R1	38.7			37.7		
	R2	42			36		

Table A5. Brookfield viscosity at 60 °C.

Modified Rotavapor aging process						
Brookfield Viscosity (Poise)						
Binder		AC-30			AC-30+10%CR		
Sample Size		50 g	125 g	200 g	50 g	125 g	200 g
Process Time	Replicate						
60 min	R1	8278	5753	4467	17929	13610	13302
	R2	7900	5188	4686	17648	14121	12938
85 min	R1	9555	6590	5185	21207	16217	14323
	R2	9745	6625	5519	20926	16952	12826
110 min	R1	12998	7841	5935	25439	17575	14107
	R2	12494	7813	6162	25838	18989	15656
Standard short-term aging processes						
Brookfield Viscosity (Poise)						
Binder		AC-30			AC-30+10%CR		
Test	Replicate						
TFOT	R1	7037			18852		
	R2	6853			18520		
RTFOT	R1	9704			25644		
	R2	9494			25883		

Table A6. Penetration at 25 °C of AC-30+12%CR.

Penetration (*0.1 mm)				
Modified Rotavapor aging process				
Process	Process	Sample Size		
Time	Temp	50 g	125 g	200 g
60 min	150 °C	45.3	46.7	48.7
	163 °C	44.2	45.3	48.7
	176 °C	42.2	44.7	46
85 min	150 °C	39.2	41.7	47
	163 °C	38.7	40.5	44
	176 °C	35	40.3	41.7
110 min	150 °C	36.8	38.7	40.3
	163 °C	35.2	38.5	39.2
	176 °C	32.7	36.7	37.7
Standard short-term aging process				
Test	Process Temp	Penetration (*0.1 mm)		
TFOT	163 °C	38		
RTFOT	163 °C	30.7		

Table A7. Brookfield viscosity at 60 °C of AC-30+12%CR.

Brookfield Viscosity (Poise)				
Modified Rotavapor aging process				
Process	Process	Sample Size		
Time	Temp	50 g	125 g	200 g
60 min	150 °C	22696	19226	17214
	163 °C	27038	19254	17458
	176 °C	28043	23782	21929
85 min	150 °C	22906	20156	18761
	163 °C	34028	26811	20739
	176 °C	41589	29723	25793
110 min	150 °C	25342	24398	19997
	163 °C	39458	31500	24862
	176 °C	51085	35424	28105
Standard short-term aging process				
Test	Process Temp	Brookfield Viscosity (Poise)		
TFOT	163 °C	29799		
RTFOT	163 °C	37399		

Table A8. Sample weight after extraction for conventional and rubber mixes.

	Sample Weight for Conventional Mixes (g)									
Time	Mix No (Left in air)									
(hour)	1	2	3	4	5	6	7	8	9	10
0	926.8	927.4	926.6	926.5	925.2	916.8	926.8	930.4	922.5	924.1
1	927.6	928.2	927.4	927.4	926.1	917.6	927.6	931.3	923.5	924.9
2	928.1	928.8	927.9	927.8	926.5	918.2	927.9	931.7	923.9	925.2
3	928.5	929	928.2	928	926.8	918.4	928.1	932	924.1	925.5
6	928.8	929.3	928.5	928.3	927.1	918.8	928.5	932.4	924.5	925.9
12	929.1	929.8	928.7	928.4	927.4	919.1	928.8	932.6	924.6	926
24	929.1	929.9	928.8	928.6	927.7	919.2	929.1	932.7	924.7	926.2
Time	Mix No (Left in dessicator)									
(hour)	1	2	3	4	5	6	7	8	9	10
0	926.8	927.4	926.6	926.5	925.2	916.8	926.8	930.4	922.5	924.1
1	926.9	927.6	926.9	926.6	925.5	917.1	927.1	930.6	922.8	924.4
2	927.1	927.7	927.0	926.8	925.7	917.2	927.3	930.7	922.9	924.5
3	927.3	927.9	927.1	926.9	925.9	917.4	927.4	930.8	923.1	924.7
6	927.4	928.2	927.2	927.2	926.0	917.5	927.5	931.0	923.2	924.7
12	927.4	928.3	927.3	927.2	926.0	917.5	927.6	931.0	923.3	924.7
24	927.4	928.2	927.3	927.1	925.9	917.5	927.6	931.0	923.3	924.7
	Sample Weight for Rubber Mixes (g)									
Time	Mix No (Left in air)									
(hour)	1	2	3	4	5	6	7	8	9	10
0	912.5	909.7	913.6	915.7	919.3	913.4	917.1	920.4	918.1	921.5
1	913.7	910.6	914.5	916.6	920.5	914.3	918.1	921.5	919.2	922.5
2	914	911.1	914.8	917	920.9	914.7	918.5	921.8	919.5	922.8
3	914.3	911.3	915.1	917.2	921.1	915	918.8	922	919.8	923.1
6	914.5	911.7	915.4	917.5	921.5	915.4	919.2	922.4	920	923.5
12	914.6	912	915.6	917.6	921.7	915.7	919.4	922.7	920.3	923.8
24	914.8	912.3	915.8	917.8	921.8	915.9	919.6	922.8	920.5	924.1
Time	Mix No (Left in dessicator)									
(hour)	1	2	3	4	5	6	7	8	9	10
0	912.5	909.9	913.6	915.6	919.4	913.4	917.0	920.4	918.1	921.5
1	912.8	910.3	913.8	916.1	919.6	913.8	917.4	920.6	918.4	921.8
2	912.9	910.4	913.9	916.1	919.7	913.8	917.5	920.8	918.5	921.9
3	913.0	910.5	914.0	916.2	919.8	914.0	917.6	920.9	918.7	922.0
6	913.1	910.6	914.1	916.3	919.9	914.2	917.7	921.0	918.8	922.1
12	913.1	910.6	914.1	916.3	920.0	914.2	917.8	921.1	918.9	922.2
24	913.1	910.6	914.1	916.3	920.0	914.2	917.8	921.1	918.9	922.2

Table A9. Computed AC content for conventional and rubber mixes.

	Computed AC content for Conventional Mixes (%)									
Time	Mix No (Left in air)									
(hour)	1	2	3	4	5	6	7	8	9	10
0	6.41	6.42	6.23	6.07	6.23	6.10	6.16	5.99	6.15	6.04
1	6.33	6.34	6.15	5.98	6.14	6.02	6.08	5.90	6.05	5.96
2	6.28	6.28	6.10	5.94	6.10	5.96	6.05	5.86	6.01	5.93
3	6.24	6.26	6.07	5.92	6.07	5.94	6.03	5.83	5.99	5.90
6	6.21	6.23	6.04	5.89	6.04	5.90	5.99	5.79	5.95	5.86
12	6.18	6.18	6.02	5.88	6.01	5.87	5.96	5.77	5.94	5.85
24	6.18	6.17	6.01	5.86	5.98	5.86	5.93	5.76	5.93	5.83
Time	Mix No (Left in dessicator)									
(hour)	1	2	3	4	5	6	7	8	9	10
0	6.41	6.42	6.23	6.07	6.23	6.10	6.16	5.99	6.15	6.04
1	6.40	6.40	6.20	6.06	6.20	6.07	6.13	5.97	6.12	6.01
2	6.38	6.39	6.19	6.04	6.18	6.06	6.11	5.96	6.11	6.00
3	6.36	6.37	6.18	6.03	6.16	6.04	6.10	5.95	6.09	5.98
6	6.35	6.34	6.17	6.00	6.15	6.03	6.09	5.93	6.08	5.98
12	6.35	6.33	6.16	6.00	6.15	6.03	6.08	5.93	6.07	5.98
24	6.35	6.34	6.16	6.00	6.16	6.03	6.08	5.93	6.07	5.98
	Computed AC content for Rubber Mixes (%)									
Time	Mix No (Left in air)									
(hour)	1	2	3	4	5	6	7	8	9	10
0	6.66	6.77	6.29	6.46	6.04	6.51	6.49	6.26	6.60	6.20
1	6.54	6.68	6.20	6.37	5.92	6.42	6.39	6.15	6.49	6.10
2	6.51	6.63	6.17	6.33	5.88	6.38	6.35	6.12	6.46	6.07
3	6.48	6.61	6.14	6.31	5.86	6.35	6.32	6.10	6.43	6.04
6	6.46	6.57	6.11	6.28	5.82	6.31	6.28	6.06	6.41	6.00
12	6.45	6.54	6.09	6.27	5.80	6.28	6.26	6.03	6.38	5.97
24	6.43	6.51	6.07	6.25	5.79	6.26	6.24	6.02	6.36	5.94
Time	Mix No (Left in dessicator)									
(hour)	1	2	3	4	5	6	7	8	9	10
0	6.66	6.75	6.29	6.47	6.03	6.51	6.50	6.26	6.60	6.20
1	6.63	6.71	6.27	6.42	6.01	6.47	6.46	6.24	6.57	6.17
2	6.62	6.70	6.26	6.42	6.00	6.47	6.45	6.22	6.56	6.16
3	6.61	6.69	6.25	6.41	5.99	6.45	6.44	6.21	6.54	6.15
6	6.60	6.68	6.24	6.40	5.98	6.43	6.43	6.20	6.53	6.14
12	6.60	6.68	6.24	6.40	5.97	6.43	6.42	6.19	6.52	6.13
24	6.60	6.68	6.24	6.40	5.97	6.43	6.42	6.19	6.52	6.13

Table A10. Temperature measurements (°C).

Vertical Distance from Oil Surface (cm)	Temp. with Morton Flask (°C)	Temp. with Standard Flask (°C)
-8	175	175
-6	175	175
-4	174	174
-2	167	161
0	151	144

Table A11. Penetration at 25 °C of residues after the modified rotavapor aging process.

Penetration in 0.1 mm				
Sample Size (g)		125	200	275
Process Time (min)	Process Temp.(°C)			
85	163	x	x	x
	176	40	x	x
110	163	38.7	x	x
		35.3	x	x
		40.8	x	x
	176	39	41	43
135	163	36.3	40	39.3
		x	38.8	x
		x	38.8	x
	176	37	40	43
160	163	34	38.7	40
		34.7	39.3	x
		35.7	38.2	x
	176	35	38	41
185	163	x	40.2	39.7
		x	35.7	33.7
		x	39.5	37.3
	176	33	36	38
210	163	x	39.7	36.2
		x	36.3	35.7
		x	39	37.5
	176	34	36	37
260	163	x	x	x
	176	30	x	x
285	163	x	x	x
	176	33	x	x
335	163	x	x	x
	176	27	x	x
435	163	28	x	x
	176	x	x	x
615	163	27	x	x
	176	x	x	x

Table A12. Viscosity at 60 °C of residues after the modified rotavapor aging process.

Brookfield Viscosity (Poise)				
Sample Size (g)		125	200	275
Process Time (min)	Process Temp.(°C)			
85	163	x	x	x
	176	47460	x	x
110	163	42217	x	x
		44109	x	x
		43527	x	x
	176	55918	40090	31932
135	163	48019	39617	34583
		x	35182	x
		x	40332	x
	176	60735	44775	34618
160	163	59901	45600	39138
		66748	41375	x
		54323	47917	x
	176	86079	50473	48943
185	163	x	48150	42444
		x	51022	63896
		x	53102	42254
	176	95346	51374	49677
210	163	x	63613	44913
		x	44218	47024
		x	54361	37471
	176	111462	66096	58676
260	163	x	x	x
	176	135585	x	x
285	163	x	x	x
	176	158611	x	x
335	163	x	x	x
	176	181497	x	x
435	163	139709	x	x
	176	x	x	x
615	163	216007	x	x
	176	x	x	x

Table A13. Dynamic modulus (G*) at 10 rad/sec and 25 °C.

Dynamic Modulus (G*) in Pascal				
Sample Size (g)		125	200	275
Process Time (min)	Process Temp.(°C)			
85	163	x	x	x
	176	x	x	x
110	163	1.14E+06	x	x
		5.11E+05	x	x
		6.87E+05	x	x
	176	x	x	x
135	163	x	1.03E+06	x
		x	1.06E+06	x
		x	9.42E+05	x
	176	x	x	x
160	163	1.39E+06	1.05E+06	x
		1.18E+06	1.32E+06	x
		1.30E+06	1.22E+06	x
	176	x	x	x
185	163	x	9.52E+05	1.04E+06
		x	1.30E+06	1.07E+06
		x	1.29E+06	9.68E+05
	176	x	x	x
210	163	x	1.36E+06	1.05E+06
		x	1.02E+06	1.17E+06
		x	1.17E+06	8.51E+05
	176	x	x	x
260	163	x	x	x
	176	x	x	x
285	163	x	x	x
	176	x	x	x
335	163	x	x	x
	176	x	x	x
435	163	1.52E+06	x	x
	176	x	x	x
615	163	2.17E+06	x	x
	176	x	x	x

Table A14. Phase angle (Delta) at 10 rad/sec and 25 °C.

Phase Angle (Delta) °				
Sample Size (g)		125	200	275
Process Time (min)	Process Temp.(°C)			
85	163	X	x	x
	176	X	x	x
110	163	50.2	x	x
	163	50.5	x	x
	163	49	x	x
	176	X	x	x
135	163	X	50.9	x
	163	X	50.9	x
	163	X	51.9	x
	176	X	x	x
160	163	49.7	51.3	x
	163	49	49.6	x
	163	49.1	49.9	x
	176	X	x	x
185	163	X	51.5	50
	163	X	49.9	47.5
	163	X	48.6	50.8
	176	X	x	x
210	163	X	49.9	48.8
	163	X	50.5	49.7
	163	X	48.6	51.1
	176	X	x	x
260	163	X	x	x
	176	X	x	x
285	163	X	x	x
	176	X	x	x
335	163	X	x	x
	176	X	x	x
435	163	45.3	x	x
	176	X	x	x
615	163	42.4	x	x
	176	X	x	x

Table A15. Dynamic modulus (G*) at 10 rad/sec and 64 °C.

Dynamic Modulus (G*) in Pascal				
Sample Size (g)		125	200	275
Process Time (min)	Process Temp.(°C)			
85	163	x	x	x
	176	x	x	x
110	163	1.07E+04	x	x
		1.06E+04	x	x
		1.08E+04	x	x
	176	x	x	x
135	163	x	8.15E+03	x
		x	9.16E+03	x
		x	8.60E+03	x
	176	x	x	x
160	163	1.19E+04	8.15E+03	x
		1.23E+04	9.60E+03	x
		1.29E+04	8.55E+03	x
	176	x	x	x
185	163	x	7.83E+03	1.14E+04
		x	1.10E+04	1.09E+04
		x	1.16E+04	9.96E+03
	176	x	x	x
210	163	x	1.14E+04	1.08E+04
		x	9.69E+03	1.07E+04
		x	1.24E+04	8.84E+03
	176	x	x	x
260	163	x	x	x
	176	x	x	x
285	163	x	x	x
	176	x	x	x
335	163	x	x	x
	176	x	x	x
435	163	2.51E+04	x	x
	176	x	x	x
615	163	3.20E+04	x	x
	176	x	x	x

Table A16. Phase Angle (Delta) at 10 rad/sec and 64 °C.

Phase Angle (Delta) °				
Sample Size (g)		125	200	275
Process Time (min)	Process Temp.(°C)			
85	163	X	X	x
	176	X	X	x
110	163	61.9	X	x
		61.3	X	x
		60.4	X	x
	176	X	X	x
135	163	X	61.2	x
		X	62.3	x
		X	62.9	x
	176	X	X	x
160	163	59.7	63.7	x
		60.3	61.8	x
		59.8	60.2	x
	176	X	X	x
185	163	X	62.7	61.6
		X	60.6	60.5
		X	60.4	61.1
	176	X	X	x
210	163	X	61.6	61.4
		X	61	60.8
		X	60	62.1
	176	X	X	x
260	163	X	X	x
	176	X	X	x
285	163	X	X	x
	176	X	X	x
335	163	X	X	x
	176	X	X	x
435	163	54.9	X	x
	176	X	X	x
615	163	53.4	X	x
	176	X	X	x

Table A17. Penetration, viscosity, G*, delta and mass change of residues after different aging processes.

Process	Penetration	Viscosity	G*	δ	G*	δ	% Mass
	at 25 °C	at 60 °C	at 25 °C	at 25 °C	at 64 °C	at 64 °C	Change
Original binder	51.7	12570	8.01E+05	55.3	5.65E+03	69.6	x
	51	11931	8.38E+05	55.2	5.17E+03	70.4	x
TFOT	37.6	43869	8.84E+05	49.6	1.17E+04	61.9	-0.1035
	38.7	43704	1.28E+06	49.6	9.36E+03	62.3	-0.0989
	x	x	x	x	x	x	-0.0964
RTFOT	35.7	52294	1.27E+06	49.6	9.87E+03	60.2	-0.10898
	36.7	63936	1.59E+06	49	1.17E+04	59.6	-0.1072
							-0.1089
TFOT+PAV	26.3	198127	2.45E+06	40.5	2.73E+04	51.8	x
	26.9	277080	2.46E+06	41	3.53E+04	52.6	x
RTFOT+PAV	28.3	204254	1.85E+06	42.1	1.41E+04	57	x
	26.3	224634	1.32E+06	46.8	1.58E+04	57.9	x
ModRvap 200g,163C,160 m	x	x	x	x	x	x	-0.1311
	x	x	x	x	x	x	-0.1244
ModRvap 200g,163C,185 m	x	x	x	x	x	x	-0.14189
	x	x	x	x	x	x	-0.13499
ModRvap 200g,163C,210 m	x	x	x	x	x	x	-0.14105
	x	x	x	x	x	x	-0.1442

Table A18. Penetration, viscosity, G*, delta and mass change of AC-20 residues after different aging processes.

Process	Penetration	Viscosity	G*	δ	G*	δ	% Mass
	at 25 °C	at 60 °C	at 25 °C	at 25 °C	at 64 °C	at 64 °C	Change
Original binder	77.2	2335	7.61E+05	71	1.29E+03	86.8	x
	78.0	2314	7.82E+05	71	1.32E+03	87.1	x
	77.8	2315	6.29E+05	71.2	1.25E+03	87.2	x
TFOT	51.7	4322	1.14E+06	65.7	2.40E+03	84.9	-0.0879
	51.7	4430	1.36E+06	66.1	2.41E+03	84.7	-0.0992
	51.5	4020	1.11E+06	66.7	2.19E+03	84.7	-0.0815
RTFOT	48.7	5319	1.42E+06	65.1	2.60E+03	84.5	-0.0941
	48.3	4809	1.43E+06	64.9	2.65E+03	84.6	-0.0998
	47.5	4401	1.38E+06	64.9	2.64E+03	84.3	-0.1038
ModRvap 200g,163C,160 m	54.0	4279	1.25E+06	66.8	2.09E+03	84.9	-0.0658
	52.7	4351	1.19E+06	66.9	1.92E+03	84.9	-0.0697
	52.0	4205	1.03E+06	67.3	2.05E+03	85.2	x
ModRvap 200g,163C,185 m	50.7	4675	1.16E+06	66.4	2.13E+03	84.8	-0.0720
	51.3	4524	1.13E+06	66.3	2.22E+03	85.2	-0.0672
	52.7	4220	1.24E+06	66.8	2.16E+03	85.1	x
ModRvap 200g,163C,210 m	48.0	5277	1.24E+06	65.7	2.40E+03	84.4	-0.0797
	47.0	5103	1.24E+06	65.5	2.44E+03	84.7	-0.0951
	48.2	5125	1.30E+06	65.4	2.04E+03	84.7	x

Table A19. Penetration, viscosity, G*, delta and mass change of AC-30 residues after different aging processes.

Process	Penetration	Viscosity	G*	δ	G*	δ	% Mass
	at 25 °C	at 60 °C	at 25 °C	at 25 °C	at 64 °C	at 64 °C	Change
Original binder	56.2	3806	8.33E+05	63.6	1.91E+03	85.3	x
	57.3	3344	6.89E+05	62.1	2.03E+03	85	x
	53.7	3431	8.88E+05	63.2	2.10E+03	85.3	x
TFOT	40.5	9302	1.29E+06	56.7	3.81E+03	81.2	-0.0548
	39.5	9160	1.83E+06	55.1	4.57E+03	80.4	-0.0544
	41.7	12692	1.44E+06	57	3.82E+03	81.7	-0.0470
	40.7	10671	1.83E+06	55.7	4.74E+03	80.1	x
	41.3	10158	1.73E+06	56.3	4.68E+03	80.8	x
	40.3	10069	1.55E+06	56.7	3.66E+03	80.3	x
	40.7	10874	1.78E+06	55.5	5.16E+03	79.9	x
	39.5	10611	1.54E+06	56.1	4.81E+03	80.2	x
	41.0	10867	1.71E+06	55.8	5.17E+03	80.3	x
	38.7	11319	1.97E+06	55.3	4.47E+03	79.9	x
RTFOT	32.8	15621	1.81E+06	53.5	5.78E+03	78.6	-0.0598
	31.5	15780	1.63E+06	54.1	5.63E+03	78.9	-0.0605
	32.2	16123	1.47E+06	53.8	6.01E+03	78.9	-0.0586
	36.3	16292	2.08E+06	53.4	6.55E+03	78.5	x
	34.5	16167	1.91E+06	54	6.36E+03	78.5	x
	34.2	16346	1.95E+06	53.5	5.91E+03	78.6	x
	34.7	16081	2.34E+06	54.3	6.40E+03	78.6	x
	36.2	15447	2.07E+06	54.2	5.69E+03	78.4	x
	38.0	15355	2.02E+06	53.9	5.55E+03	78.8	x
	36.3	16032	2.16E+06	53.8	6.30E+03	78.3	x

Table A19 Continued

Process	Penetration	Viscosity	G*	δ	G*	δ	% Mass
	at 25 °C	at 60 °C	at 25 °C	at 25 °C	at 64 °C	at 64 °C	Change
ModRvap	38.0	11131	1.34E+06	54.7	4.64E+03	80.7	-0.0674
200g,163C,160 m	37.8	13407	1.83E+06	55.2	5.20E+03	79.6	-0.0687
	37.0	11713	1.13E+06	55.8	4.66E+03	80.5	x
	37.2	11200	1.74E+06	55.8	4.83E+03	81.1	x
	37.7	11450	1.81E+06	55.7	5.03E+03	80.2	x
	38.7	11646	1.55E+06	56.1	4.74E+03	80.8	x
	38.3	11245	1.81E+06	56.4	5.03E+03	80.5	x
	34.2	12254	1.38E+06	56.4	4.72E+03	80.5	x
	34.7	11709	1.62E+06	55.9	4.68E+03	80.8	x
	33.8	11842	1.48E+06	56.7	4.80E+03	80.4	x
ModRvap	35.7	12745	1.52E+06	55.6	4.73E+03	80.5	-0.0813
200g,163C,185 m	36.8	11355	1.39E+06	53.5	4.68E+03	80.8	-0.0673
	40.8	11665	1.74E+06	55.5	4.60E+03	80.6	x
	36.2	13883	1.80E+06	55	5.21E+03	79.8	x
	33.2	13359	1.81E+06	55.4	5.53E+03	79.8	x
	32.8	13300	1.39E+06	55.2	4.82E+03	79.9	x
	32.7	13479	1.75E+06	54.9	5.68E+03	79.8	x
	33.3	13347	1.82E+06	55.2	5.37E+03	79.8	x
	33.5	13945	1.70E+06	55.4	5.37E+03	79.6	x
	33.8	13516	1.71E+06	55.3	5.42E+03	79.3	x
ModRvap	33.8	14782	1.99E+06	53.3	5.54E+03	79.4	-0.0822
200g,163C,210 m	35.0	14773	1.69E+06	53.5	5.51E+03	79.3	-0.0753
	34.3	13162	1.71E+06	53.2	5.41E+03	79.6	x
	31.5	15476	1.54E+06	54.3	4.47E+03	79.1	x
	32.7	15768	1.84E+06	54.3	5.78E+03	79.4	x
	31.7	15600	1.86E+06	54.4	6.73E+03	78.4	x
	30.7	15739	1.91E+06	54.1	6.45E+03	78.8	x
	31.7	17534	2.08E+06	53.2	6.34E+03	78.5	x
	32.2	15044	1.87E+06	54.4	5.61E+03	79.4	x
	31.0	16394	1.95E+06	54	6.03E+03	78.5	x

Table A20. Penetration, viscosity, G*, delta and mass change of AC-30+5%CR residues after different aging processes.

Process	Penetration	Viscosity	G*	δ	G*	δ	% Mass
	at 25 °C	at 60 °C	at 25 °C	at 25 °C	at 64 °C	at 64 °C	Change
Original binder	50.3	11434	1.11E+06	53	3.67E+03	75	x
	53.2	10152	7.01E+05	57.8	3.50E+03	75.7	x
	53.3	10548	9.08E+05	57.2	3.54E+03	76.4	x
TFOT	39.2	25504	1.36E+06	52.8	7.11E+03	69.8	-0.0928
	39.2	27551	1.13E+06	53.5	6.96E+03	69.8	-0.0826
	37.0	23445	9.86E+05	53.9	7.14E+03	69.8	-0.1071
	41.3	22489	1.35E+06	53.4	6.83E+03	71.1	x
	38.5	21083	1.11E+06	53.1	6.30E+03	71.5	x
	41.0	23969	1.16E+06	53.8	6.16E+03	71.7	x
	42.5	20893	1.24E+06	54	6.84E+03	69.8	x
	40.0	20296	1.28E+06	53.6	6.24E+03	71.5	x
	42.0	22152	1.27E+06	53.8	6.85E+03	70	x
	42.7	24846	1.20E+06	53.5	6.73E+03	69.5	x
RTFOT	33.7	25119	1.32E+06	52.5	8.22E+03	69.6	-0.0633
	35.3	27297	1.26E+06	51.8	8.22E+03	68.3	-0.0748
	34.0	23462	1.18E+06	53	8.84E+03	69.5	-0.0743
	33.0	23897	1.29E+06	52.9	8.01E+03	69.9	x
	32.8	27298	1.42E+06	51.8	8.06E+03	68.6	x
	31.8	24025	1.41E+06	52.8	6.82E+03	71.1	x
	33.7	21761	1.04E+06	55	8.20E+03	70.2	x
	33.3	23817	1.22E+06	53.3	6.95E+03	71.1	x
	34.2	27297	1.19E+06	52.4	7.00E+03	70.4	x
	31.8	24333	1.29E+06	53.2	7.95E+03	70.2	x

Table A20 Continued

Process	Penetration	Viscosity	G*	δ	G*	δ	% Mass
	at 25 °C	at 60 °C	at 25 °C	at 25 °C	at 64 °C	at 64 °C	Change
ModRvap	36.5	22001	1.08E+06	53.3	6.90E+03	69.6	-0.0858
200g,163C,160 m	38.5	21184	1.05E+06	53.6	6.41E+03	70.9	-0.0906
	39.3	17607	9.16E+05	55.4	5.46E+03	72.8	x
	37.5	23651	1.27E+06	53.1	7.23E+03	69.5	x
	38.2	20556	1.14E+06	53.6	7.13E+03	69.4	x
	38.2	23925	1.24E+06	53.3	7.39E+03	69.2	x
	38.3	18417	1.09E+06	53.5	7.11E+03	70.1	x
	41.2	19881	1.38E+06	53.4	7.29E+03	69.3	x
	38.7	23051	1.26E+06	54	7.48E+03	69.4	x
	38.7	21987	1.38E+06	53.6	7.12E+03	69.5	x
ModRvap	35.7	20674	1.16E+06	52.8	7.25E+03	69.8	-0.0828
200g,163C,185 m	37.7	26746	1.19E+06	52.9	7.42E+03	69.8	-0.0834
	35.7	25553	1.05E+06	52.4	7.92E+03	69	x
	37.2	17256	1.23E+06	53.2	7.63E+03	68.3	x
	35.8	25576	1.33E+06	53	7.69E+03	69.6	x
	35.0	26743	1.24E+06	52.8	7.38E+03	70	x
	37.3	23381	1.32E+06	53	7.74E+03	69.5	x
	37.3	26697	1.28E+06	52.6	8.17E+03	67.9	x
	36.7	19953	1.46E+06	53	7.67E+03	69.5	x
	38.2	25173	1.23E+06	52.8	7.98E+03	68.1	x
ModRvap	35.3	28022	1.34E+06	52.2	7.32E+03	69.4	-0.0839
200g,163C,210 m	35.7	17327	1.24E+06	52	7.86E+03	69.8	-0.0911
	35.0	27772	1.20E+06	51.8	7.69E+03	68.6	x
	36.0	29000	1.26E+06	52.1	8.54E+03	68.1	x
	35.3	28001	1.28E+06	51.8	8.63E+03	68.4	x
	34.3	17635	1.21E+06	52.4	8.62E+03	68	x
	35.8	26400	1.25E+06	52.4	8.77E+03	67.5	x
	34.2	29273	1.37E+06	52	8.52E+03	68.1	x
	34.5	27915	1.40E+06	52.1	8.99E+03	67.9	x
	35.2	27695	1.36E+06	51.9	8.32E+03	68.2	x

Table A21. Penetration, viscosity, G*, delta and mass change of AC-30+12%CR residues after different aging processes.

Process	Penetration	Viscosity	G*	δ	G*	δ	% Mass
	at 25 °C	at 60 °C	at 25 °C	at 25 °C	at 64 °C	at 64 °C	Change
Original binder	51.7	12570	8.01E+05	55.3	5.65E+03	69.6	x
	51.0	11931	8.38E+05	55.2	5.17E+03	70.4	x
TFOT	37.7	41507	8.84E+05	49.6	1.17E+04	61.9	-0.1035
	35.5	47695	1.28E+06	49.6	9.36E+03	62.3	-0.0989
	38.0	42315	6.06E+05	61.3	2.42E+03	80.4	-0.0964
RTFOT	36.7	49395	1.27E+06	49.6	8.64E+03	60.9	-0.1090
	35.7	49299	1.59E+06	49	9.87E+03	60.2	-0.1072
	35.3	59240	1.38E+06	49	1.17E+04	59.6	-0.1089
ModRvap 200g,163C,160 m	38.7	45600	1.05E+06	51.3	8.15E+03	63.7	-0.1311
	39.3	41375	1.23E+06	50.7	9.60E+03	61.8	-0.1244
	38.2	47917	1.22E+06	49.9	8.55E+03	60.2	x
ModRvap 200g,163C,185 m	40.2	48150	9.52E+05	51.5	7.83E+03	62.7	-0.1419
	35.7	51022	1.30E+06	49.9	1.10E+04	60.6	-0.1350
	39.5	53102	1.29E+06	48.6	1.16E+04	60.4	x
ModRvap 200g,163C,210 m	39.7	63613	1.36E+06	49.9	1.14E+04	61.6	-0.1442
	36.3	44218	1.12E+06	50.3	9.69E+03	61	-0.1411
	39.0	54361	1.17E+06	48.6	1.24E+04	60	x

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BIOGRAPHICAL SKETCH

Okan Sirin was born on December 16, 1971, in Urfa, Turkey. He attended elementary, secondary, and high schools in Izmir, Turkey. He received his high school diploma from Karsiyaka Gazi High School in 1988.

After graduating from high school, he was admitted to Dokuz Eylul University. He graduated as the third best among the civil engineering graduates and received a bachelor of science degree in civil engineering from Dokuz Eylul University in Izmir, Turkey in July 1992.

After succeeding in an exam given by the Ministry of Education in Turkey, he was awarded a scholarship to carry out his graduate study in the USA. He attended the LCP International Institute in Sacramento, CA, to improve his English from June 1993 to December 1993.

In January 1994, he started his graduate study at the University of Illinois at Urbana-Champaign. He received his master of science degree in civil engineering in January 1996.

In August 1996, he transferred to the University of Florida and started his doctoral program under the supervision of Dr. Mang Tia in the civil engineering materials group.
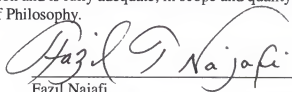
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Mang Tia, Chair
Professor of Civil Engineering

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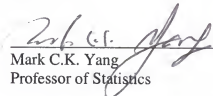
Byron E. Ruth
Professor of Civil Engineering

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Reynaldo Roque
Professor of Civil Engineering

I certify that I have read this study and that in my opinion it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation for the degree of Doctor of Philosophy.

Fazil Najafi
Associate Professor of Civil Engineering

I certify that I have read this study and that in my opinion it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation for the degree of Doctor of Philosophy.

Mark C.K. Yang
Professor of Statistics

This dissertation was submitted to the Graduate Faculty of the College of Engineering and to the Graduate School and was accepted as partial fulfillment of the requirements for the degree of Doctor of Philosophy.

August, 2000

M. Jack Ohanian
Dean, College of Engineering

Winfred M. Phillips
Dean, Graduate School